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OFFICIAL AND TENTATIVE

METHODS OF ANALYSIS

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

AS COMPILED BY THE COMMITTEE ON REVISION OF METHODS
(R. E. DOOLITTLE, Chairman, B. L. HARTWELL, G. W. HOOVER,
A. J. PATTEN, A. F. SEEKER AND W. A. WITHERS)

WITH AN INTRODUCTION BY
HARVEY W. WILEY
HONORARY PRESIDENT OF THE ASSOCIATION

Revised to November 1, 1919

4040

PUBLISHED BY THE
ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS
AT WASHINGTON, D. C.
SEPTEMBER, 1920

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PREFACE

In presenting this revision of the official and tentative methods of analysis of the Association of Official Agricultural Chemists, it is appropriate to give a brief statement of the organization of the association, its purpose and the procedure by which the methods are adopted.

Membership in the Association is institutional and includes the State Departments of Agriculture, the State Agricultural Colleges and Experiment Stations, the Federal Department of Agriculture and the Federal, State and City offices charged with the enforcement of food, feed, drug, fertilizer, insecticide and fungicide control laws.

The Association was founded at Philadelphia, Pa., September 9, 1884, by the following representative agricultural chemists of that time, the organization being the result of a series of informal meetings held the immediately preceding years:

Prof. H. W. Wiley, Chemist of the Department of Agriculture, Washington, D. C.
Mr. Clifford Richardson, Assistant Chemist of the Department of Agriculture, Washington, D. C.
Mr. Philip E. Chazal, State Chemist of South Carolina.
Dr. Chas. W. Dabney, Jr., State Chemist of North Carolina.
Dr. J. W. Gascoyne, State Chemist of North Carolina.
Dr. E. H. Jenkins, Connecticut Experiment Station.
Prof. John A. Myers, State Chemist of Mississippi.
Prof. H. C. White, State Chemist of Georgia.
Mr. C. DeGhequier, Secretary National Fertilizer Association.
Dr. Schumann, Dr. Lehmann, Mr. Gaines and others.

At the first meeting methods for the determination of phosphoric acid and potash in commercial fertilizers were adopted and work was begun for the perfection and adoption of methods for the entire range of agricultural chemistry. Later the passage of food and drug and insecticide and fungicide control legislation by the states and by the Federal Government made it necessary to extend the scope of the Association's activities for reason that the Association methods were designated as the official methods for the enforcement of such legislation as well as for the control of feeds and fertilizers by the various states.

To attain the aims of the association for a set of accurate methods, a system was evolved by which the methods in question are subjected to the most rigorous and painstaking scrutiny before they can be adopted. A "referee" is appointed for any subject for which the association has not yet an official method or for a method which seems to require further investigation. The referee conducts analyses according to the methods suggested for adoption in comparison with methods already established, obtaining the collaboration of as many as possible of the workers in that field. In addition, a great deal of original research has been inaugurated on new methods. This system developed logically until at the present time, in order to be adopted as "tentative", a method must be recommended to the association by the referee, and such recommendation is made only after the method has undergone a thorough collaborative and critical study. Further, the special committee on methods must approve the recommendation and the method must be accepted by a vote of the association. In order to become "official", a method must be again accepted at another annual meeting. The recommendations of referees are published in the reports of the proceedings of the association in the *Journal of the Association of Official Agricultural Chemists*, so that all tentative methods are made public before being adopted. This permits consideration and criticism by chemists who are not members of the association. It is immediately apparent that a method can be made official only after the most thorough series of tests, not alone for accuracy, but for ease of operation as well. It may be stated without reservation that more elaborate and painstaking effort has been expended on this collection of analytical methods than upon any other set of similar methods in the field of chemical science.

The compilation and revision of the methods presented in this book was made by a committee of the association, consisting of R. E. Doolittle, chairman; B. L. Hartwell, G. W. Hoover, A. F. Seeker (deceased), J. P. Street and W. A. Withers. Later, on the resignation of J. P. Street, A. J. Patten was appointed a member of the committee and the work of revision was continued.

A preliminary revision, antedating the revision published in this book, was printed in 1916 as supplementary parts to Volumes I and II of the *Journal of the Association of Official Agricultural Chemists*. In this preliminary revision the committee received important assistance from R. L. Emerson, F. C. Blanck and N. A. Parkinson. At that time the scheme of numbering the sections in each chapter was adopted in order to simplify the system of cross-references.

In the preparation of the present revision J. A. MacLaughlin rendered valuable assistance. Acknowledgment is also made to the Library of the Department of Agriculture for assistance.

Throughout its work, it has been the aim of the committee not only to bring the methods up to date, but especially to state the procedure with such lucidity and in such detail as to make it possible for any trained chemist to operate without being in doubt at any time.

The work of the committee has been one of critical revision, compilation and editing. The work of developing the methods was done by the various referees and their collaborators who have reported to the association at its annual meetings during the last decade. To them is due the credit for the subject matter of this book.

C. L. Alsberg,
Secretary of the Association of
Official Agricultural Chemists.

September 17, 1920.

INTRODUCTION

By Dr. Harvey W. Wiley, Honorary President of the Association of Official Agricultural Chemists.

In the present edition of the Methods of Analysis, official and tentative, of the Association of Official Agricultural Chemists, the technique of analytical procedures has been revised to November 1, 1919. The monumental work of the Association of Official Agricultural Chemists is not only well known in the profession in this country, but is recognized in all countries as being the last word in agricultural chemical technique. The methods of determining the composition of agricultural products, as well as of all bodies related to agriculture, has been recognized also by the courts of this country. In case of judicial proceedings where different methods of analysis have been employed, the court, in all cases where the question has arisen, has recognized the official methods as binding.

At the time of the organization of this body, referred to in the Preface, agricultural methods of research, from the chemical point of view, were extremely chaotic. The progress of agricultural science which has marked its history in the last third of a century could not have been maintained amid these chaotic conditions. The methods adopted by the founders of this association for correcting this state of affairs have been shown by experience to be the best possible. I can say that the improvement in agricultural chemical technique has almost kept pace with the growth of the association.

The gradual incorporation in the membership of the association of those scientific men engaged in the control of foods and drugs has widened the scope without altering the purpose of the original founders. Today we find a body of scientific workers in agriculture and related subjects numbering quite half a thousand, who, by their activities and collaboration, have contributed to the pages of this volume, directly or indirectly. The scientific knowledge of agriculture which has been verified and extended by this association now forms the foundation of all agricultural improvement.

The profession of agriculture is the fundamental industry of this country. Everything which strengthens the foundations of this industry benefits the country at large. Our workers are not banded together for personal preferment, either in wages or in authority. They have united for the sole purpose of benefitting agriculture and thus increasing production. They have not asked for shorter hours, nor for higher pay. They have worked in season, out of season, by day and by night, on work days and holidays, to perfect that science which, in its application, is the most powerful factor in scientific agriculture.

The ability of the agricultural industry to withstand the assaults which are made upon it at the present time is largely due to the successful efforts of our association. The agricultural industry has been built upon a rock and thus it is able to withstand the winds and the floods. This industry is now in a more critical condition than any other. The allurements of the city and the high wages of labor therein, have drawn from the farm much of its best blood and energy. Congregate life has become so much more attractive than discrete life that it is hard to keep the young of both sexes upon the farm. Yet it is plain that if man power and woman power upon the farm now be depleted the industry must suffer. Making the farm attractive does not merely mean beautifying the house in which the farmer lives, making it more sanitary, planting trees, flowers and shrubs, but it means also the best knowledge of the soil and its properties; the most scientific data respecting the manufacture and use of fertilizing materials; the most accurate knowledge of the character of crops best suited to the soil and the best system of rotation which will help develop from the soil its most generous contribution. In other words, not only must the farmer's farm be attractive and sanitary, but it must also be productive and dividend paying.

We can well imagine the worth of the work which our association has done by picturing for a moment what the present agricultural industry would be if all that our science has contributed to it were stricken from human records. In such a deplorable condition starvation would surely be staring the world in the face. In the quiet corners of the laboratory, by the midnight oil and by personal devotion, the means which enable the farmer to get more remunerative crops have been worked out and perfected. These workers, male and female, who have done this gigantic task have never been heralded in the public press, nor received encomiums of an admiring world. They have done their work silently and effectively, without expectation of praise and without hope of pecuniary reward. Their real reward has been in the consciousness of duty done. The referees who have presided over this great work for the past thirty-six years and those who have aided them in these investigations, merit the generous regard and esteem of the whole scientific world, as well as the whole agricultural world. Our association has been not one of debate nor of visionary plans of human welfare, but rather of hard work and consecrated devotion to the cause.

The volume which is now laid before you contains the very last word of all that is important in agricultural research from the chemical and physical point of view. This does not mean that the field is fully exploited. The great unknown of tomorrow doubtless holds in its secret embrace even greater prospects for human betterment than the days which have already passed. This association stands ready and with expectant breath to receive the messages of tomorrow and translate them to the agricultural world.

Washington, D. C.,
September 15, 1920.

CONTENTS

PAGE

Preface iii

Introduction. By Dr. Harvey W. Wiley v

CHAPTER

I. Fertilizers 1

II. Inorganic Plant Constituents 15

III. Waters 21

IV. Tanning Materials 43

V. Leathers 49

VI. Insecticides and Fungicides 53

VII. Foods and Feeding Stuffs 71

VIII. Saccharin Products 101

IX. Food Preservatives 117

X. Coloring Matters in Foods 131

XI. Metals in Foods 147

XII. Fruits and Fruit Products 153

XIII. Canned Vegetables 161

XIV. Cereal Foods 167

XV. Wines 173

XVI. Distilled Liquors 181

XVII. Beers 187

XVIII. Vinegars 191

XIX. Flavoring Extracts 197

XX. Meat and Meat Products 209

XXI. Dairy Products 225

XXII. Fats and Oils 239

XXIII. Spices and Other Condiments 257

XXIV. Cacao Products 265

XXV. Coffees 269

XXVI. Teas 273

XXVII. Baking Powders and Baking Chemicals 277

XXVIII. Drugs 291

XXIX. Soils 209

XXX. Reference Tables 321

ILLUSTRATIONS

PAGE.
Fig. 1. Method of Filling Combustion Tube for Nitrogen Determination........ 9
Fig. 2. Metal Extractor Used for Extracting Tanning Materials.............. 46
Fig. 3. Apparatus for Distillation of Arsenious Chlorid.................... 54
Fig. 4. Apparatus for Determination of Formic Acid......................... 129
Fig. 5. Apparatus for Determination of Arsenic.............................. 148
Fig. 6. Apparatus for Determination of Volatile Acids...................... 177
Fig. 7. Apparatus for Ammonia Determination................................. 210
Fig. 8. Van Slyke Apparatus for Determination of Amino Nitrogen............ 217
Fig. 9. Details of the Deaminizing Bulb and Connection..................... 218
Fig. 10. Apparatus for Melting Point Determination......................... 243
Fig. 11. Apparatus for Determination of Polenske Number.................... 249
Fig. 12. Knorr's Apparatus for Determination of Carbon Dioxid.............. 277
Fig. 13. Heidenhain's Apparatus for Determination of Carbon Dioxid......... 279
Fig. 14. Apparatus for Electrolytic Determination of Lead.................. 287
Fig. 15. Quadruplicate Shaking Apparatus for Determination of Carbonate Carbon in Soils.. 310
Fig. 16. Absorption Tower for Carbon Dioxid................................ 311
Fig. 17. Single Apparatus for Determination of Carbonate Carbon............ 811
Fig. 18. Parr's Apparatus for Determination of Carbon Dioxid............... 313

*REFERENCE TABLES

	TABLE NUMBER.	PAGE.
Alcohol Table, for calculating the percentages of alcohol in mixtures of ethyl alcohol and water from their specific gravities	7	345
for calculating the percentages of alcohol in mixtures of ethyl alcohol and water from their Zeiss immersion refractometer readings at 17.5°–25°C.	8	361
Corrections, for temperature, in densities of cane sugar solutions	4	342
in conjunction with Geerlig's Table	6	344
to readings of saccharometers standard at 20°C.	9	388
Degrees Brix, specific gravity and degrees Baumé of sugar solutions	10	389
Densities of solutions of cane sugar at 20°C.	3	338
Dextrose (Munson and Walker's Table)	1	321
Dry substance, in sugar house products, by the Abbé refractometer at 28°C., Geerlig's Table	5	343
Ethyl alcohol, in mixtures with water, by specific gravity	7	345
by Zeiss immersion refractometer	8	361
Geerlig's Table, for dry substance in sugar house products, by the Abbé refractometer at 28°C.	5	343
Invert Sugar (Munson and Walker's Table)	1	321
Kröber's Table, for determining pentoses and *pentosans*	2	332
Lactose (Munson and Walker's Table)	1	321
Maltose (Munson and Walker's Table)	1	321
Munson and Walker's Table, for dextose, invert sugar, lactose and maltose	1	321
Pentoses and pentosans, Kröber's Table	2	332
Specific gravity, degrees Brix and degrees Baumé, of sugar solutions	10	389

***The term "water" used throughout this book indicates "distilled water".**

The abbreviation A. O. A. C. is recognized as official for the name of the Association of Official Agricultural Chemists. For this reason the letters J. A. O. A. C. are used in all bibliographies in referring to the Journal of the Association of Official Agricultural Chemists.

Official and Tentative Methods of Analysis

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

(REVISED TO NOVEMBER 1, 1919)

I. FERTILIZERS.

GENERAL METHODS.

1 MECHANICAL ANALYSIS OF BONE AND TANKAGE.—OFFICIAL.

Transfer 100 grams of the original material to a sieve having circular openings $\frac{1}{50}$ inch (0.5 mm.) in diameter. Sift, breaking the lumps by means of a soft rubber pestle if the material has a tendency to cake. Weigh the coarse portion remaining on the sieve. Determine the fine portion by difference.

2 PREPARATION OF SAMPLE.—OFFICIAL.

Reduce the gross sample by quartering to an amount sufficient for analytical purposes. Transfer to a sieve having circular openings $\frac{1}{25}$ inch (1 mm.) in diameter, sift, breaking the lumps with a soft rubber pestle. Grind in a mortar the part remaining on the sieve until the particles will pass through. Mix thoroughly and preserve in tightly stoppered bottles. Grind and sift as rapidly as possible to avoid loss or gain of moisture during the operation.

It is recommended that the gross sample be taken by means of a sampler which removes a core from the top to the bottom of the bag; that at least a pound of the material shall constitute an official sample sent to the laboratory; and that the entire sample submitted to the chemist be passed through a 10 mesh sieve previous to its subdivision for analysis.

3 MOISTURE.—OFFICIAL.

Heat 2 grams of the sample prepared as in **2** for 5 hours in a water oven at the temperature of boiling water. In the case of potash salts, sodium nitrate and ammonium sulphate, heat at about 130°C. to constant weight. The loss in weight is considered as moisture.

TOTAL PHOSPHORIC ACID.

Gravimetric Method.—Official.

4 REAGENTS.

(a) *Molybdate solution.*—Dissolve 100 grams of molybdic acid in dilute ammonium hydroxid (144 cc. of ammonium hydroxid (sp. gr. 0.90) and 271 cc. of water); pour this solution slowly and with constant stirring into dilute nitric acid (489 cc. of nitric acid (sp. gr. 1.42) and 1148 cc. of water). Keep the mixture in a warm place for several days or until a portion heated to 40°C. deposits no yellow precipitate of ammonium phosphomolybdate. Decant the solution from any sediment and preserve in glass-stoppered vessels.

(b) *Ammonium nitrate solution.*—Dissolve 200 grams of commercial ammonium nitrate, phosphate free, in water and dilute to 2 liters.

(c) *Magnesia mixture.*—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding an excess of the acid. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, aluminium, and phosphoric acid; filter; add 280 grams of ammonium chlorid, 261 cc. of ammonium hydroxid (sp. gr. 0.90) and dilute to 2 liters. Instead of the solution of 22 grams of calcined magnesia, 110 grams of crystallized magnesium chlorid ($MgCl_2 6H_2O$) dissolved in water may be used, then add 280 grams of ammonium chlorid and proceed as above.

(d) *Dilute ammonium hydroxid for washing.*—Dilute 100 cc. of ammonium hydroxid (sp. gr. 0.90) to 1 liter.

(e) *Magnesium nitrate solution.*—Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess, boil, filter from the excess of magnesia, ferric oxid, etc., and dilute to 2 liters.

5 PREPARATION OF SOLUTION.

Treat 2 grams of the sample by one of the methods given below. In the case of (d) 2.5 grams may be used. Cool the solution, dilute to 200 cc., or 250 cc. if a 2.5 gram sample was used. Mix and pour on a dry filter.

(a) Ignite, and dissolve in hydrochloric acid.

(b) Evaporate with 5 cc. of magnesium nitrate, ignite, and dissolve in hydrochloric acid.

(c) Boil with 20–30 cc. of strong sulphuric acid in a Kjeldahl flask, adding 2–4 grams of sodium or potassium nitrate at the beginning of the digestion and a small quantity after the solution has become nearly colorless, or adding the nitrate in small portions from time to time. After the solution is colorless add 150 cc. of water and boil for a few minutes.

(d) Digest in a Kjeldahl flask with strong sulphuric acid and such other reagents as are used in either the plain or modified Kjeldahl or Gunning method for estimating nitrogen. Do not add potassium permanganate, but after the solution has become colorless add about 100 cc. of water and boil for a few minutes.

(e) Dissolve in 30 cc. of concentrated nitric acid and a small quantity of hydrochloric acid and boil until organic matter is destroyed.

(f) Add 30 cc. of concentrated hydrochloric acid, heat and add cautiously, in small quantities at a time, about 0.5 gram of finely pulverized potassium chlorate to destroy organic matter.

(g) Dissolve in 15–30 cc. of strong hydrochloric acid and 3–10 cc. of nitric acid. This method is recommended for fertilizers containing much iron or aluminium phosphate.

6 DETERMINATION.

Take an aliquot of the solution prepared as directed above, corresponding to 0.25 gram, 0.50 gram, or 1 gram, neutralize with ammonium hydroxid and clear with a few drops of nitric acid. In case hydrochloric or sulphuric acid has been used as a solvent, add about 15 grams of dry ammonium nitrate or a solution containing that amount. To the hot solution add 70 cc. of the molybdate solution for every decigram of phosphoric acid (P_2O_5) present. Digest at about 65°C. for an hour, and determine if the phosphoric acid has been completely precipitated by the addition of more molybdate solution to the clear supernatant liquid. Filter and wash with cold water or, preferably, ammonium nitrate solution. Dissolve the precipitate on the filter with ammonium hydroxid and hot water and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and from a burette add slowly (about 1 drop per second), stirring vigorously, 15 cc. of magnesia mixture for each decigram of phosphoric acid (P_2O_5) present. After 15 minutes add 12 cc. of ammonium hydroxid (sp. gr. 0.90). Let stand till the supernatant liquid is clear (2 hours is usually enough), filter, wash with the dilute ammonium hydroxid until the washings are practically free from chlorids, ignite to whiteness or to a grayish white, weigh and calculate to phosphoric acid (P_2O_5).

Volumetric Method.—Official.

7 REAGENTS.

(a) *Molybdate solution.*—To 100 cc. of molybdate solution prepared as directed in **4** **(a)**, add 5 cc. of nitric acid (sp. gr. 1.42). This solution should be filtered immediately before using.

(b) *Standard sodium or potassium hydroxid solution.*—Dilute 323.81 cc. of N/1 alkali, free from carbonates, to 1 liter. One hundred cc. of the solution should neutralize 32.38 cc. of N/1 acid; 1 cc. is equivalent to 1 mg. of P_2O_5 (1% of P_2O_5 on a basis of 0.1 gram of substance).

(c) *Standard acid solution.*—Prepare an acid solution corresponding to the strength of **(b)**, or to one-half of this strength, and standardize by titration against that solution, using phenolphthalein as indicator. Hydrochloric or nitric acid may be used.

(d) *Phenolphthalein solution.*—Dissolve 1 gram of phenolphthalein in 100 cc. of alcohol.

8 PREPARATION OF SOLUTION.

Dissolve according to **5** **(b)**, **(e)**, **(f)**, or **(g)**, preferably by **(e)**, when these acids are a suitable solvent, and dilute to 200 cc. with water.

9 DETERMINATION.

(a) For percentages up to 5 use an aliquot corresponding to 0.4 gram of substance, for percentages between 5 and 20 use an aliquot corresponding to 0.2 gram of substance, and for percentages above 20 use an aliquot corresponding to 0.1 gram of substance. Add 5–10 cc. of nitric acid, depending on the method of solution (or the equivalent in ammonium nitrate), nearly neutralize with ammonium hydroxid, dilute to 75–100 cc., heat in a water bath to 60°–65°C., and for percentages below 5 add 20–25 cc. of freshly filtered molybdate solution. For percentages between 5 and 20 add 30–35 cc. of molybdate solution. For percentages greater than 20 add sufficient molybdate solution to insure complete precipitation. Stir, let stand in the bath about 15 minutes, filter *at once*, wash once or twice with water by decantation, using 25–30 cc. each time, agitate the precipitate thoroughly and allow to settle; transfer to the filter and wash with cold water until the filtrate from 2 fillings of the filter yields a pink color upon the addition of phenolphthalein and 1 drop of the standard alkali. Transfer the precipitate and filter to the beaker or precipitating vessel, dissolve the precipitate in a small excess of the standard alkali, add a few drops of phenolphthalein solution and titrate with the standard acid.

(b) Proceed as in **(a)** with this exception: Heat in a water bath at 45°–50°C., add the molybdate solution, and allow to remain in the bath with occasional stirring for 30 minutes.

(c) Proceed as in **(a)** to the point where the solution is ready to place in the water bath. Then cool the solution to room temperature, add molybdate solution at the rate of 75 cc. for each decigram of phosphoric acid present, place the stoppered flask containing the solution in a shaking apparatus and shake for 30 minutes at room temperature, filter at once, wash, and titrate as in (**a**).

WATER-SOLUBLE PHOSPHORIC ACID.

10 *Gravimetric Method.—Official.*

Place 2 grams of the sample on a 9 cm. filter, wash with successive small portions of water, allowing each portion to pass through before adding more, until the filtrate measures about 250 cc. If the filtrate is turbid, add a little nitric acid. Make up to any convenient volume, mix well, use an aliquot and proceed as directed under **6**.

11 *Volumetric Method.—Official.*

Treat the sample as directed under **10**. To an aliquot of the solution corresponding to 0.2 or 0.4 gram, add 10 cc. of concentrated nitric acid, nearly neutralize with ammonium hydroxid, dilute to 60 cc. and proceed as directed under **9**.

CITRATE-INSOLUBLE PHOSPHORIC ACID.—OFFICIAL.

12 REAGENTS.

In addition to the reagents described under **4** and **7** prepare ammonium citrate solution by one of the following methods:

Ammonium citrate solution.—(1) Dissolve 370 grams of commercial citric acid in 1500 cc. of water; nearly neutralize with commercial ammonium hydroxid; cool; add ammonium hydroxid until exactly neutral to corallin (saturated alcoholic solution), and dilute sufficiently to make the specific gravity 1.09 at 20°C. The volume will be about 2 liters; or,

(2) To 370 grams of commercial citric acid add commercial ammonium hydroxid until nearly neutral; reduce the specific gravity to slightly more than 1.09 at 20°C. and make exactly neutral, testing as follows: Prepare a solution of fused calcium chlorid, 200 grams to the liter, and add 4 volumes of strong alcohol. Make this solution exactly neutral, using a small amount of freshly prepared corallin solution as preliminary indicator, and test finally by withdrawing a portion, diluting with an equal volume of water, and testing with cochineal solution; 50 cc. of this solution will precipitate the citric acid from 10 cc. of the citrate solution. To 10 cc. of the nearly neutral citrate solution add 50 cc. of the alcoholic calcium chlorid solution, stir well, filter at once through a folded filter, dilute with an equal volume of water and test the reaction with neutral solution of cochineal. If acid or alkaline, add ammonium hydroxid or citric acid, as the case may be, to the citrate solution, mix and test again, as before. Repeat this process until a neutral reaction is obtained. Add sufficient water to make the specific gravity 1.09 at 20°C.

13 DETERMINATION.

(**a**) *Acidulated samples.*—Heat 100 cc. of strictly neutral ammonium citrate solution (sp. gr. 1.09) to 65°C. in a 250 cc. flask placed in a warm water bath, keeping the flask loosely stoppered to prevent evaporation. The level of the water in the bath should be above that of the citrate solution in the flask. When the citrate solution has reached 65°C., drop into it the filter containing the washed residue from the water-soluble phosphoric acid solution in **10**, close tightly with a smooth rubber stopper and shake violently until the filter paper is reduced to a pulp, relieving the pressure by momentarily removing the stopper. Place the flask in the bath and maintain its contents at exactly 65°C. Shake the flask every 5 minutes. At the expiration of exactly 30 minutes from the time the filter and the residue are introduced, remove the flask from the bath and immediately filter the contents as rapidly as possible, through a quick-acting filter paper. Wash with water at 65°C. until the volume of the filtrate is about 350 cc., allowing time for thorough draining before adding new portions of water. (1) Transfer the filter and its contents to a crucible, ignite until all organic matter is destroyed, add 10–15 cc. of strong hydrochloric acid and digest until all phosphate is dissolved; or, (2) Return the filter with contents to the digestion flask, add 30–35 cc. of strong nitric acid, 5–10 cc. of strong hydrochloric acid and boil until all phosphate is dissolved. Dilute the solution as prepared in (1) or (2) to 200 cc. If desired, the filter and its contents may be treated according to method **5** (**b**), (**c**) or (**d**). Mix well, filter through a dry filter and proceed as directed under **6** or **9**.

(**b**) *Non-acidulated samples.*—In case a determination of citrate-insoluble phosphoric acid is required in non-acidulated samples treat 2 grams of the phosphatic material without previous washing with water, precisely as in (**a**), except when the substance contains much animal matter (bone, fish, etc.), in which case dissolve the residue insoluble in ammonium citrate by any one of the processes described under **5** (**b**), (**c**) or (**d**) and determine phosphoric acid as directed under **6** or **9**.

14 CITRATE-SOLUBLE PHOSPHORIC ACID.—OFFICIAL.

The sum of the water-soluble and citrate-insoluble subtracted from the total gives the citrate-soluble phosphoric acid.

15 DETECTION OF NITRATES.—OFFICIAL.

Mix 5 grams of the fertilizer with 25 cc. of hot water and filter. To a portion of this solution add 2 volumes of concentrated sulphuric acid, free from nitric acid and oxids of nitrogen and allow the mixture to cool. Add cautiously a few drops of a concentrated solution of ferrous sulphate so that the fluids do not mix. If nitrates are present the junction shows at first a purple, afterwards a brown, color or if only a very minute quantity be present, a reddish color. To another portion of the solution add 1 cc. of a 1 per cent solution of sodium nitrate and test as before to determine whether sufficient sulphuric acid was added in the first test.

ORGANIC AND AMMONIACAL NITROGEN ONLY.

Kjeldahl Method.—Official.

16 REAGENTS.

For ordinary work N/2 acid is recommended. For work in determining very small amounts of nitrogen N/10 acid is recommended. In titrating mineral acids against ammonium hydroxid solution use cochineal or methyl red as indicator.

(**a**) *Standard hydrochloric acid.*—Determine the absolute strength as follows: *Preliminary test.*—Place a measured portion of the acid to be standardized in an Erlenmeyer flask with excess of calcium carbonate to neutralize free acid and a few drops of potassium chromate as indicator. By titration with silver nitrate solution determine exactly the quantity required to precipitate the chlorids. *Final determination.*—To a measured portion of the acid to be standardized add from the burette 1 drop in excess of the required quantity of silver nitrate solution as determined by the preceding test. Heat to boiling, protect from the light, and allow to stand until the precipitate is granular. Filter on a tared Gooch crucible, previously heated to 140°–150°C., wash with hot water, testing the filtrate to prove excess of silver nitrate. Dry the silver chlorid at 140°–150°C., cool and weigh.

(b) *Standard sulphuric acid.*—Determine the absolute strength of the acid by precipitation with barium chlorid solution as follows: Dilute a measured quantity of the acid to be standardized to approximately 100 cc., heat to boiling and add drop by drop a 10 per cent solution of barium chlorid until no further precipitation occurs. Continue the boiling for about 5 minutes, allow to stand for 5 hours or longer in a warm place, pour the supernatant liquid on a tared Gooch crucible or an ashless filter, treat the precipitate with 25–30 cc. of boiling water, transfer to the filter and wash with boiling water until the filtrate is free from chlorids. Dry, ignite over a Bunsen burner and weigh as barium sulphate.

(c) *Standard alkali solution.*—Accurately determine the strength of this solution by titration against the standard acid. N/10 solution is recommended.

(d) *Sulphuric acid.*—Sp. gr. 1.84 and free from nitrates and ammonium sulphate.

(e) *Metallic mercury, or mercuric oxid.*—Mercuric oxid should be prepared in the wet way, but not from mercuric nitrate.

(f) *Copper sulphate.*—Crystallized.

(g) *Granulated zinc or pumice stone.*—Added to the contents of the distillation flask if necessary to prevent bumping.

(h) *Potassium sulphid solution.*—Dissolve 40 grams of commercial potassium sulphid in 1 liter of water.

(i) *Sodium hydroxid solution.*—A saturated solution, free from nitrates.

(j) *Cochineal solution.*—Digest, with frequent agitation, 3 grams of pulverized cochineal in a mixture of 50 cc. of strong alcohol and 200 cc. of water for 1 or 2 days at ordinary temperature, and then filter.

(k) *Methyl red solution.*—Dissolve 1 gram of methyl red (dimethyl-amino-azo-benzene-ortho-carboxylic acid) in 100 cc. of 95 per cent alcohol.

17 APPARATUS.

(a) *Kjeldahl flasks for both digestion and distillation.*—Total capacity of about 550 cc., made of hard, moderately thick, and well-annealed glass.

(b) *Distillation flasks.*—For distillation any suitable flask of about 550 cc. capacity may be used. It is fitted with a rubber stopper through which passes the lower end of a Kjeldahl connecting bulb to prevent sodium hydroxid being carried over mechanically during distillation. The bulb should be about 3 cm. in diameter, and the tubes should be of the same diameter as the condenser tube with which the upper end of the bulb tube is connected by means of rubber tubing.

18 DETERMINATION.

Place 0.7–3.5 grams, according to the nitrogen content, of the substance to be analyzed in a digestion flask with approximately 0.7 gram of mercuric oxid, or its equivalent in metallic mercury, and add 20–30 cc. of sulphuric acid (0.1–0.3 gram of crystallized copper sulphate may also be used in addition to the mercury, or in place of it). Place the flask in an inclined position and heat below the boiling point of the acid until frothing has ceased. (A small piece of paraffin may be added to prevent extreme foaming.) Then increase the heat until the acid boils briskly and digest for a time after the mixture is colorless or nearly so, or until oxidation is complete.

After cooling dilute with about 200 cc. of water, add a few pieces of granulated zinc or pumice stone, if necessary to prevent bumping, and 25 cc. of potassium sulphid solution with shaking (when no mercury or mercuric oxid is used the addition of potassium sulphid is unnecessary). Next add sufficient sodium hydroxid solution to make the reaction strongly alkaline, 50 cc. are usually enough, pouring it down the side of the flask so that it does not mix at once with the acid solution. Connect the flask with the condenser, mix the contents by shaking, distil until all ammonia has passed over into a measured quantity of the standard acid and titrate with the standard alkali. The first 150 cc. of the distillate will generally contain all the ammonia.

Previous to use the reagents should be tested by a blank experiment with sugar. The sugar partially reduces any nitrates present that might otherwise escape notice.

Gunning Method.—Official.

19

REAGENTS.

Potassium sulphate, or anhydrous sodium sulphate.—Pulverized.

The other reagents and standard solutions used are described under **16**.

20

APPARATUS.

The apparatus used is described under **17**.

21

DETERMINATION.

Place 0.7–3.5 grams, according to the nitrogen content, of the substance to be analyzed in a digestion flask. Add 10 grams of powdered potassium sulphate, or 10 grams of anhydrous sodium sulphate, and 15–25 cc. (ordinarily about 20 cc.) of sulphuric acid (0.1–0.3 gram of crystallized copper sulphate may also be added). Conduct the digestion as in the Kjeldahl process, starting with a temperature below the boiling point and increasing the heat gradually until frothing ceases. Digest for a time after the mixture is colorless or nearly so, or until oxidation is complete. Complete the determination as directed under **18** except that no potassium sulphid is added. In making alkaline before distilling it is convenient to add litmus paper or a few drops of phenolphthalein indicator. The pink color given by phenolphthalein indicating an alkaline reaction is destroyed by a considerable excess of strong fixed alkali.

Kjeldahl-Gunning-Arnold Method.—Official.

22

REAGENTS AND APPARATUS.

Described under **16**, **17** and **19**.

23

DETERMINATION.

Place 0.7–3.5 grams, according to the nitrogen content, of the substance to be analyzed in a digestion flask. Add 15–18 grams of potassium sulphate, or 15–18 grams of anhydrous sodium sulphate, 1 gram of copper sulphate, 1 gram of mercuric oxid, or its equivalent in metallic mercury, and 25 cc. of sulphuric acid. Heat gently until frothing ceases, then boil the mixture briskly, and continue the digestion for a time after the mixture is colorless or nearly so or until oxidation is complete. Cool, dilute with about 200 cc. of water, add 50 cc. of potassium sulphid solution, make strongly alkaline with sodium hydroxid solution and complete the determination as directed under **18**.

TOTAL NITROGEN.

Kjeldahl Method Modified to include the Nitrogen of Nitrates.—Official.

24

REAGENTS.

(**a**) *Zinc dust.*—Impalpable powder. Granulated zinc or zinc filings will not answer.

(**b**) *Sodium thiosulphate.*

(**c**) *Commercial salicylic acid.*

The other reagents and standard solutions are described under **16**.

25

APPARATUS.

The apparatus used is described under **17**.

26

DETERMINATION.

Place 0.7–3.5 grams, according to the nitrogen content, of the substance to be analyzed in a Kjeldahl digestion flask. (1) Add 30 cc. of sulphuric acid containing 1 gram of salicylic acid, shake until thoroughly mixed, allow to stand for at least 30 minutes and then add 5 grams of crystallized sodium thiosulphate and digest as directed below; or, (2) Add to the substance 30 cc. of sulphuric acid containing 2 grams of salicylic acid, allow to stand at least 30 minutes and then add gradually 2 grams of zinc dust, shaking the contents of the flask at the same time, and digest as follows:

Place the flask on the stand for holding the digestion flasks and heat over a low flame until all danger from frothing has passed. Then increase the heat until the acid boils briskly and continue the boiling until white fumes no longer escape from the flask. This requires about 5–10 minutes. Add approximately 0.7 gram of mercuric oxid, or its equivalent in metallic mercury, and continue the boiling until the liquid in the flask is colorless, or nearly so. In case the contents of the flask are likely to become solid before this point is reached, add 10 cc. more of sulphuric acid. Complete the determination as directed under **18**. The reagents should be tested by blank experiments.

Gunning Method Modified to include the Nitrogen of Nitrates.—Official.

27

REAGENTS AND APPARATUS.

The apparatus, reagents and standard solutions are described under **16**, **17**, **19** and **24**.

28

DETERMINATION.

Place 0.7–3.5 grams, according to the nitrogen content, of the substance to be analyzed in a digestion flask. Add 30–35 cc. of salicylic acid mixture (1 gram of salicylic acid to 30 cc. of sulphuric acid); shake until thoroughly mixed and allow to stand for at least 30 minutes with frequent shaking. Add 5 grams of sodium thiosulphate and heat the solution for 5 minutes; cool; add 10 grams of potassium sulphate, or 10 grams of anhydrous sodium sulphate, heat very gently until foaming ceases and proceed as directed under **21**.

Absolute or Cupric Oxid Method.—Official.

29

REAGENTS.

(**a**) *Coarse cupric oxid.*—Ignite and cool before using.

(**b**) *Fine cupric oxid.*—Grind (**a**).

(**c**) *Metallic copper.*—Granulated copper, or fine copper gauze, heated and cooled in a current of hydrogen or by dropping the heated copper into a test tube containing a few cc. of methyl alcohol.

(**d**) *Sodium bicarbonate.*—Free from organic matter.

(**e**) *Caustic potash solution.*—A supersaturated solution of caustic potash in hot water.

30

APPARATUS.

(**a**) *Combustion tube.*—Hard Bohemian glass, about 65 cm. long, 12.7 mm. internal diameter and sealed at one end.

(**b**) *Azotometer.*—Capacity 100 cc., accurately calibrated.

(**c**) *Sprengel mercury air pump.*

(**d**) *Small paper scoop.*—Made from stiff writing paper.

31 DETERMINATION.

Use 1–2 grams of ordinary commercial fertilizers. In the case of highly nitrogenized substances, the amount to be used is governed by the amount of nitrogen estimated to be present. Fill the tube (Fig. 1) as follows: (1) about 5 cm. of coarse cupric oxid; (2) place on the small paper scoop an amount of the fine cupric oxid which, when mixed with the substance to be analyzed, will fill about 10 cm. of the tube; pour on this the substance, rinsing the watch glass with a little of the fine oxid, and mix thoroughly with a spatula, pour into the tube, rinsing the scoop with a little fine oxid; (3) about 30 cm. of coarse cupric oxid; (4) about 7 cm. of metallic copper; (5) about 6 cm. of coarse cupric oxid; (6) a small plug of asbestos; (7) 0.8–1 gram of sodium bicarbonate; (8) a large loose plug of asbestos.

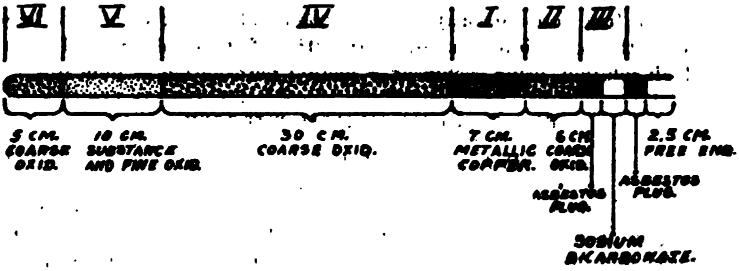

FIG. I. COMBUSTION TUBE FOR THE DETERMINATION OF TOTAL NITROGEN.

The Roman numerals refer to the order in which the different portions are to be heated.

After the tube is filled hold in a horizontal position and tap gently on the table in order that a canal may be formed in the upper portion of the fine cupric oxid. Place the tube in the combustion furnace, leave about 2.5 cm. of it projecting and connect with the pump by a rubber stopper smeared with glycerol, taking care to make the connection perfectly tight. In order to protect the latter from the heat, place an asbestos plate, having a circular opening in the center, over the projecting end of the tube.

Exhaust the air from the tube by means of the pump. When a vacuum has been obtained, allow the flow of mercury to continue; light the gas under that part of the tube containing the metallic copper, the anterior layer of cupric oxid and the sodium bicarbonate. As soon as the vacuum is destroyed and the apparatus filled with carbon dioxid, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer just below the surface of the mercury seal so that the escaping bubbles will pass into the air and not into the tube, to avoid the useless saturation of the caustic potash solution.

When the flow of carbon dioxid has very nearly or completely ceased, pass the delivery tube down into the receiving arm so that the bubbles will escape into the azotometer. Light the gas under the 30 cm. layer of oxid, heat gently for a few minutes, to drive out any moisture that may be present, and then bring to a red heat. Heat gradually the mixture of substance and oxid, lighting a jet at a time. Avoid a too rapid evolution of bubbles, which should be allowed to escape at the rate of about one per second or a little faster. When the burners under the mixture have all been turned on, light the gas under the layer of oxid at the end of the tube. When the evolution of bubbles has ceased, turn out all the burners except those under the metallic copper and anterior layer of oxid, and allow to cool for a few minutes. Exhaust with the pump and remove the azotometer before the flow of mercury has stopped. Break the connection of the tube with the pump, stop the flow of mercury and extinguish the burners. Allow the azotometer to stand for at least an hour, or cool with a stream of water until the volume and temperature become constant.

Adjust accurately the level of the potassium hydroxid solution in the bulb to that in the azotometer; note the volume of the nitrogen, temperature and height of barometer. Calculate the weight of the nitrogen as usual.

AMMONIACAL NITROGEN.

32

Magnesium Oxid Method.—Official.

Place 0.7–3.5 grams, according to the ammonia content, of the substance to be analyzed in a distillation flask with about 200 cc. of water and 5 grams or more of magnesium oxid, free from carbonates. Then connect the flask with a condenser and distil 100 cc. of the liquid into a measured quantity of standard acid and titrate with standard alkali solution, using cochineal or methyl red solution as indicator.

NITRIC AND AMMONIACAL NITROGEN.

33

Reduced Iron Method.—Official.

Place 1 gram of the sample in a 500 cc. flask, add about 30 cc. of water and 2–3 grams of reduced iron, and, after standing sufficiently long to insure solution of the soluble nitrates and ammonium salts, add 10 cc. of a mixture of strong sulphuric acid with an equal volume of water; shake thoroughly, place a long-stemmed funnel in the neck of the flask to prevent mechanical loss and allow to stand for a short time until the violence of the reaction has moderated. Heat the solution slowly, boil for 5 minutes and cool. Add about 100 cc. of water, a little paraffin, and 7–10 grams of magnesium oxid, free or nearly free from carbonates. Connect with a condenser, such as is used in the Kjeldahl method and boil the mixture for 40 minutes, nearly to dryness; collect the ammonia in a measured quantity of standard acid and titrate with standard alkali solution in the usual manner. The nitrogen obtained represents the nitrates plus the ammonium salts contained in the sample.

In the analysis of nitrate salts proceed as above, except that 25 cc. of the nitrate solution, equivalent to 0.25 gram of the sample, are employed with 5 grams of reduced iron. After boiling add 75 cc. of water and an excess of sodium hydroxid solution and complete the determination as above.

34

Zinc-Iron Method.—Official.

Dissolve 10 grams of the sample in water and dilute to 500 cc. Place 25 cc. of this solution, corresponding to 0.5 gram of the substance, in a 400 cc. distillation flask, add 120 cc. of water, 5 grams of well-washed and dried zinc dust and 5 grams of reduced iron. To the solution add 80 cc. of saturated sodium hydroxid solution, connect the flask with the condensing apparatus and conduct the distillation simultaneously with the reduction, collecting the ammonia in standard acid. Continue the distillation until 100 cc. have been distilled and titrate with standard alkali, using cochineal or methyl red solution as indicator.

NITROGEN IN NITRATE SALTS.

35

Ferrous Sulphate-Zinc-Soda Method.—Official.

Place 0.5 gram of the nitrate salt in a 600–700 cc. flask, add 200 cc. of water, 5 grams of powdered zinc, 1–2 grams of ferrous sulphate and 50 cc. of sodium hydroxid solution (36° Baumé). Connect with the distilling apparatus, distil, collect the distillate in the usual way in N/10 sulphuric acid and titrate with standard alkali, using cochineal or methyl red solution as indicator.

WATER-INSOLUBLE ORGANIC NITROGEN SOLUBLE IN NEUTRAL PERMANGANATE.—OFFICIAL.

36 *Preliminary Test (Determination of Water-Insoluble Organic Nitrogen).*

Place 1 gram of the material on an 11 cm. filter paper and wash with water at room temperature until the filtrate measures 250 cc. Dry and determine nitrogen in the residue as in **18** or **21**, making a correction for the nitrogen of the filter, if necessary.

37 DETERMINATION.

Place a quantity of the fertilizer, equivalent to 50 mg. of water-insoluble organic nitrogen as determined in **36**, on a moistened 11 cm. filter paper and wash with water at room temperature until the filtrate measures 250 cc. Transfer the insoluble residue with 25 cc. of tepid water to a 300 cc. Griffin low-form beaker, add 1 gram of sodium carbonate, mix and add 100 cc. of 2 per cent permanganate solution. Cover with a watch glass and immerse for 30 minutes in a steam or hot water bath so that the level of the liquid in the beaker is below that of the water in the bath. Stir twice at intervals of 10 minutes. At the end of the digestion remove from the bath, add immediately 100 cc. of cold water and filter through a heavy 15 cm. folded filter. Wash with small quantities of cold water until the filtrate measures about 400 cc. Determine nitrogen in the residue and filter, as in **18** or **21**, correcting for the nitrogen contained in the latter. The nitrogen thus obtained is the inactive water-insoluble organic nitrogen. Subtract this result from that obtained in **36** to obtain the water-insoluble organic nitrogen soluble in neutral permanganate.

WATER-INSOLUBLE ORGANIC NITROGEN DISTILLED FROM ALKALINE PERMANGANATE.—OFFICIAL.

38 PREPARATION OF SAMPLE.

(**a**) *Mixed fertilizers.*—Place an amount of material, equivalent to 50 mg. of water-insoluble organic nitrogen determined as directed under **36**, on a filter paper and wash with water at room temperature until the filtrate measures 250 cc.

(**b**) *Raw materials.*—Place an amount of material equivalent to 50 mg. of water-insoluble organic nitrogen, determined as directed under **36**, in a small mortar, add about 2 grams of powdered rock phosphate, mix thoroughly, transfer to a filter paper and wash with water at room temperature until the filtrate measures 250 cc. When much oil or fat is present, it is well to wash with ether before extracting with water.

39 DETERMINATION.

Dry the residue remaining after treatment of the material as described under **38** at a temperature not exceeding 80°C. and transfer from the filter to a 500–600 cc. Kjeldahl distillation flask. Add 20 cc. of water, 15–20 small glass beads, or fragments of pumice stone, a piece of paraffin the size of a pea and 100 cc. of alkaline permanganate solution (25 grams of pure potassium permanganate and 150 grams of sodium hydroxid, separately dissolved in water, the solutions cooled, mixed, and made to a volume of 1 liter). Connect with an upright condenser to the lower end of which a receiver containing standard acid has been attached. Digest slowly, for at least 30 minutes, below distillation point, with a very low flame, using coarse wire gauze and asbestos paper between the flask and flame. Gradually raise the temperature and, after any danger from frothing has passed, distil until 95 cc. of the distillate are obtained, and titrate as usual. When a tendency to froth is noticed, lengthen the digestion period and no trouble will be experienced when the distillation is begun. During the digestion gently rotate the flask occasionally, particularly if the material shows a tendency to adhere to the sides. The nitrogen thus obtained is the active water-insoluble organic nitrogen.

POTASH.

Lindo-Gladding Method.—Official.

40 REAGENTS.

(**a**) *Ammonium chlorid solution.*—Dissolve 100 grams of ammonium chlorid in 500 cc. of water, add 5–10 grams of pulverized potassium platinic chlorid and shake at intervals for 6–8 hours. Allow the mixture to settle overnight and filter. The residue may be used for the preparation of a fresh supply.

(**b**) *Platinum solution.*—A platinic chlorid solution containing the equivalent of 1 gram of metallic platinum (2.1 grams of H_2PtCl_6) in every 10 cc.

(**c**) 80 *per cent alcohol.*—Sp. gr. 0.8593 at $\frac{20^\circ C.}{4^\circ}$

41 PREPARATION OF SOLUTION.

(**a**) *Mixed fertilizers.*—Place 2.5 grams of the sample upon a 12.5 cm. filter paper and wash into a 250 cc. graduated flask with successive small portions of boiling water until the filtrate amounts to about 200 cc. Add to the hot solution a slight excess of ammonium hydroxid and sufficient ammonium oxalate to precipitate all the lime present, cool, dilute to 250 cc., mix, and pass through a dry filter.

(**b**) *Potash salts; muriate and sulphate of potash, sulphate of potash and magnesia, and kainit.*—Dissolve 2.5 grams and dilute to 250 cc. without the addition of ammonium hydroxid and ammonium oxalate.

(**c**) *Organic compounds.*—When it is desired to determine the total amount of potash in organic substances, such as cottonseed meal, tobacco stems, etc., saturate 10 grams of the sample with strong sulphuric acid and ignite in a muffle at a low red heat to destroy organic matter. Add a little strong hydrochloric acid, warm slightly in order to loosen the mass from the dish, transfer to a 500 cc. graduated flask, add ammonium hydroxid and ammonium oxalate, cool, dilute to 500 cc., mix, pass through a dry filter and proceed as directed under **42** (**a**).

(**d**) *Ashes from wood, cotton hulls, etc.*—Boil 10 grams of the sample with 300 cc. of water for 30 minutes, add to the hot solution a slight excess of ammonium hydroxid and then sufficient ammonium oxalate to precipitate all the lime present. Cool, dilute to 500 cc., mix and pass through a dry filter.

42 DETERMINATION.

(**a**) *Mixed fertilizers.*—Evaporate a 50 cc. aliquot of the solution **41** (**a**) nearly to dryness, add 1 cc. of dilute sulphuric acid (1 to 1), evaporate to dryness and ignite to whiteness. Maintain a full red heat until the residue is perfectly white. Dissolve the residue in hot water, using at least 20 cc. for each decigram of potassium oxid present, add a few drops of hydrochloric acid and platinum solution in excess. Evaporate on a water bath to a thick paste. Treat the residue with 80 per cent alcohol, avoiding exposure to ammonia. Filter, wash the precipitate thoroughly with 80 per cent alcohol both by decantation and on the filter, continuing the washing after the filtrate is colorless.

Then wash with 10 cc. of the ammonium chlorid solution to remove impurities from the precipitate and repeat 5 or 6 times. Wash again thoroughly with 80 per cent alcohol and dry the precipitate for 30 minutes at 100°C. Weigh and calculate to potassium oxid. The precipitate should be perfectly soluble in water.

(**b**) *Muriate of potash.*—Acidify 50 cc. of the solution prepared according to **41** (**b**) with a few drops of hydrochloric acid, add 10 cc. of platinum solution and evaporate to a thick paste. Treat the residue as directed under (**a**).

(**c**) *Sulphate of potash; sulphate of potash and magnesia; and kainit.*—Acidify 50 cc. of the solution prepared according to **41** (**b**) with a few drops of hydrochloric acid and add 15 cc. of platinum solution. Evaporate the mixture and proceed as directed under (**a**), except that 25 cc. portions of the ammonium chlorid solution should be used.

(**d**) *Water-soluble potash in ashes from wood, cotton hulls, etc.*—Prepare the solution according to **41** (**d**) and proceed as directed under (**a**), paying special attention to the last sentence.

For the conversion of potassium platinic chlorid to potassium chlorid use the factor 0.3067; to potassium sulphate, 0.3585; to potassium oxid, 0.1938.

Alternative Method.—Official.

(The Lindo-Gladding method is preferable in the presence of soluble sulphates.)

43 REAGENTS.

Described under **40**.

44 PREPARATION OF SOLUTION.

Prepare the solution as directed under **41**, omitting in all cases the addition of ammonium hydroxid and ammonium oxalate.

45 DETERMINATION.

Dilute 25 cc. of the solution made as directed under **44** (50 cc. if less than 10% of potassium oxid be present) to 150 cc., heat to 100°C., and add, drop by drop, with constant stirring, a slight excess of barium chlorid solution. Without filtering, add in the same manner barium hydroxid solution in slight excess. Filter while hot and wash until the precipitate is free from chlorids. Add to the filtrate 1 cc. of strong ammonium hydroxid, and then a saturated solution of ammonium carbonate until the excess of barium is precipitated. Heat and add, in fine powder, 0.5 gram of pure oxalic acid or 0.75 gram of ammonium oxalate. Filter, wash free from chlorids, evaporate the filtrate to dryness in a platinum dish and ignite carefully over the free flame, below a red heat, until all volatile matter is driven off. Digest the residue with hot water, filter through a small filter and dilute the filtrate, if necessary, so that for each decigram of potassium oxid there will be at least 20 cc. of liquid. Acidify with a few drops of hydrochloric acid and add platinum solution in excess. Evaporate on a water bath to a thick paste and treat the residue with 80 per cent alcohol, both by decantation and after collecting on a Gooch or other form of filter, dry for 30 minutes at 100°C. and weigh. If there is an appearance of foreign matter in the double salt, it should be washed as in **42** (**a**) with several portions of 10 cc. each of the ammonium chlorid solution.

THOMAS OR BASIC SLAG.

46 MECHANICAL ANALYSIS.—OFFICIAL.

Proceed as directed under **1**, using 10 grams of material.

47 PREPARATION OF SAMPLE.—OFFICIAL.

Prepare the sample as directed under **2**.

TOTAL PHOSPHORIC ACID.

Gravimetric Method.—Official.

48 PREPARATION OF SOLUTION.

Prepare the solution for analysis as directed under **5** (**g**), or in strong hydrochloric acid alone. In the latter case after the portion for analysis is measured out, add nitric acid and heat for a few minutes.

49 DETERMINATION.

Dehydrate an aliquot (20 cc.) of the solution obtained as directed under **48** by evaporating to dryness on a steam or hot water bath; treat with 5 cc. of hydrochloric acid and 25 cc. of hot water; digest in order to complete solution and filter off silica. From this point proceed as directed under **6**. Before precipitating with magnesia mixture, add 5 cc. of 5 per cent sodium acetate solution.

50 *Volumetric Method.—Official.*

Prepare the solution as directed under **5** (**g**) and determine the phosphoric acid in an aliquot of this solution as directed under **9**, standardizing the solutions against a standard phosphate material of approximately the same composition as the sample under examination.

CITRATE-SOLUBLE PHOSPHORIC ACID.

Gravimetric Method.—Tentative.

51 PREPARATION OF SOLUTION.

Weigh 5 grams of the slag into a 500 cc. Wagner flask containing 5 cc. of 95 per cent alcohol. (The flask should have a neck width of at least 22 mm. and should be marked at least 8 cm. below the mouth.) Make up to the mark with 2 per cent citric acid solution of a temperature of 17.5°C. Fit the flask with a rubber stopper and place at once in a rotary apparatus, shaking the flask for 30 minutes at the rate of 30–40 revolutions per minute, at the end of which time remove the flask, filter immediately on a dry filter and analyze the solution at once.

52 DETERMINATION.

To 50 cc. of the clear filtrate in a beaker add 100 cc. of molybdate solution prepared as directed under **4** (**a**). Place the beaker in a water bath, until the temperature of the contents reaches 65°C., remove from the bath and cool to room temperature. Filter and wash the yellow precipitate of ammonium phosphomolybdate 4 or 5 times with 1 per cent nitric acid. Dissolve the precipitate in 100 cc. of cold 2 per cent ammonium hydroxid, nearly neutralize with hydrochloric acid and add to the solution, drop by drop, with continuous stirring, 15 cc. of magnesia mixture prepared as directed under **4** (**c**), and proceed as under **6**.

53 *Volumetric Method.—Tentative.*

In an aliquot of the clear solution, prepared as in **51**, determine the phosphoric acid as directed under **9**.

II. INORGANIC PLANT CONSTITUENTS.

1 PREPARATION OF SAMPLE.—OFFICIAL.

Thoroughly cleanse the material from all foreign matter, especially from adhering soil, air-dry, grind, and preserve the sample in tightly stoppered bottles.

2 PREPARATION OF ASH.—OFFICIAL.

Ignite 10–20 grams of the substance, in a flat-bottomed platinum dish in a muffle, at a comparatively low temperature. Do not employ a full red heat because of the danger of volatilizing alkali chlorids, etc. If rich in silica and alkalies, char the material, treat with water to dissolve soluble salts, filter through an ashless filter, dry the filter and incinerate, add the filtrate to the incinerated residue, evaporate to dryness and ignite at a low red heat. If rich in phosphates, *e. g.*, seeds and animal substances, char the material, dissolve soluble salts in dilute acetic acid, filter through an ashless filter, wash with water, dry and incinerate the filter and residue, add the filtrates to the incinerated residue, evaporate to dryness and ignite gently. While still warm, pulverize the whole ash as obtained above, mix intimately and preserve in a tightly stoppered, dry bottle. If after incineration the ash has absorbed moisture, dry thoroughly at low redness before bottling.

3 CARBON DIOXID.—OFFICIAL.

Determine carbon dioxid in a weighed portion of the ash as prepared under **2**. Liberate the carbon dioxid by treatment with dilute hydrochloric acid in any of the usual forms of apparatus, and determine the increase in weight of the potash bulbs. The efficiency of the apparatus should be tested by blank determinations conducted upon weighed portions of pure calcite.

4 CARBON, SAND AND SILICA.—OFFICIAL.

Transfer the residue from the carbon dioxid determination to a beaker or evaporating dish; evaporate to dryness; pulverize and dry thoroughly to render the silica insoluble. Moisten the dry residue with 5–10 cc. of hydrochloric acid, add about 50 cc. of water, allow to stand on a water bath for a few minutes, filter through a hardened filter and wash thoroughly. Dilute the solution and washings to 250 cc. or other convenient volume. Designate as *A*.

Wash the residue from the filter into a platinum dish and boil for about 5 minutes with approximately 20 cc. of a saturated solution of pure sodium carbonate, add a few drops of pure sodium hydroxid solution, allow the solution to settle and decant through a tared Gooch filter. Boil the residue in the dish with sodium carbonate solution and decant as before. Repeat the process again, then transfer the residue to the Gooch filter, wash thoroughly, first with hot water, then with a little dilute hydrochloric acid, and finally with hot water until free from chlorids. Dry the filter and contents to constant weight at 110°C. to determine the combined weight of carbonaceous material and sand. Incinerate; the loss in weight represents the carbonaceous material; the residue is sand. Confirm by microscopic examination. Determine the soluble silica as follows: (1) Combine the alkaline filtrate and washings, acidify with hydrochloric acid, evaporate to dryness and determine the silica in the usual way; or, (2) Treat a weighed portion of the ash, as prepared under **2**, with dilute hydrochloric acid. Evaporate to dryness; pulverize and dry thoroughly to render the silica insoluble. Moisten the dry residue with 5–10 cc. of hydrochloric acid, add about 50 cc. of water, allow to stand on the water bath for a few minutes, filter on an ashless filter, wash, dry, ignite and weigh to determine the combined weight of the silica and sand. Deduct the weight of the sand found above to obtain that of the silica. The soluble silica can not be separated from the residue after ignition.

5 CARBON-FREE ASH.—OFFICIAL.

Subtract the weights of the carbon found in **4** and the carbon dioxid found in **3** from that of the total ash used in **3**.

6 FERRIC AND ALUMINIUM OXIDS.—OFFICIAL.

(Applicable for plant materials other than seeds.)

Pipette an aliquot of *A*, under **4**, corresponding to 0.5 gram of ash, into a 250 cc. beaker. If ferrous iron is present, oxidize it by boiling with a few cc. of hydrogen peroxid or of concentrated nitric acid. Cool, add ammonium hydroxid until a precipitate begins to form, then nitric acid until just clear, and finally 2–3 cc. of concentrated nitric acid in excess. Add 25 cc. of 50 per cent ammonium nitrate solution, phosphate free, heat to 40°C., and add slowly, with constant shaking, a moderate excess of molybdate solution [I, 4 (a)], and allow to stand for 1–2 hours at a temperature not exceeding 40°C. After standing for an hour pipette 5 cc. of the clear solution into an equal volume of warm molybdate solution. If a precipitate forms in the test portion return it to the original solution and add more molybdate solution. Allow to stand at room temperature for several hours, preferably overnight. Filter, wash with about 75 cc. of ammonium nitrate solution (2.5%, phosphate free, and slightly acidified with nitric acid) and combine the filtrate and washings. Designate as *B*. Reserve the precipitate for the determination of phosphoric acid as described under **11**.

Without concentrating solution *B*, cautiously neutralize with ammonium hydroxid, add a very slight excess of the alkali, avoiding a temperature higher than 40°C., and allow to stand at this temperature until the precipitate completely settles. Filter the clear supernatant liquid, wash the precipitate a few times by decantation with hot water before transferring to the filter, then wash 4 or 5 times on the filter. Dissolve the precipitate on the filter with hot nitric acid (1 to 5), wash and reprecipitate as before. The combined filtrates and washings from the first and second precipitations should not exceed 500 cc. and should not be concentrated by evaporation. Designate as *C* and reserve for the determination of calcium and magnesium as described under **8**. The same filter may be used for the second filtration, and the volume of the solution for the reprecipitation need not exceed 100 cc. Before the second filtration is made, a small quantity of ashless filter paper pulp should be added in order to facilitate washing and leave the precipitate finely divided after ignition, so that it can be easily fused with potassium hydrogen sulphate for the iron determination. Dry and ignite the precipitate and weigh as ferric and aluminium oxids.

Determine the iron oxid in the following manner: Fuse, in a platinum crucible, the ignited precipitate with about 4 grams of fused potassium hydrogen sulphate. This fusion takes but a few minutes and must not be continued beyond the time actually needed. When complete set the crucible aside and allow to cool. Add 5 cc. of concentrated sulphuric acid and heat until copious fumes of sulphuric acid are given off. Cool, transfer to a flask, add water and digest till the solution is clear. Reduce with zinc, cool, titrate with N/50 potassium permanganate and calculate to ferric oxid.

If it is desired to use a larger amount of the sample for the iron determination, evaporate a suitable aliquot of *A*, under **4**, with sulphuric acid, reduce with zinc and titrate as above.

MANGANESE, CALCIUM AND MAGNESIUM.

7 *Method I.—Official.*

(Applicable for plant materials other than seeds.)

Manganese.—To an aliquot of *A*, under **4**, corresponding to 0.5–2 grams of ash, add a quantity of pure ferric chlorid solution, more than sufficient to combine with the phosphoric acid which may be present, and neutralize with ammonium hydroxid. Dissolve the precipitate in a very slight excess of hydrochloric acid and add 1–2 grams of sodium acetate. Boil for 1–2 minutes, filter at once, and wash with boiling water. Dissolve the precipitate in hydrochloric acid and reprecipitate as above. Concentrate the combined filtrates and washings to about 50 cc., cool, add bromin water until the solution is colored, make alkaline with ammonium hydroxid, and heat to boiling in a covered beaker; cool, and repeat the addition of bromin water, of ammonium hydroxid and boil again. If a precipitate is obtained, slightly acidify the solution with acetic acid, filter immediately, and wash with hot water. Dry the precipitate, ignite and weigh as manganomanganic oxid (Mn_3O_4).

Calcium.—Concentrate the filtrate and washings from the manganese determination to about 50 cc., make slightly alkaline with ammonium hydroxid, and add, while still hot, ammonium oxalate solution, drop by drop, slightly in excess of complete precipitation, to convert the magnesium also into oxalate. Heat to boiling, allow the precipitate to settle completely, decant the clear solution on a filter, pour 15–20 cc. of hot water on the precipitate, and again decant the clear solution on the filter. Dissolve the precipitate in the beaker with a few drops of hydrochloric acid, add a little water, repeat the precipitation as above, and filter through the same filter; transfer the precipitate to the filter and wash free from chlorids with hot water; dry, ignite the precipitate over the blast lamp to constant weight, and weigh as calcium oxid.

Magnesium.—Evaporate the combined filtrates and washings from the calcium determination to dryness on a water bath and heat carefully to expel ammonium salts. Treat the residue with 20–25 cc. of hot water and about 5 cc. of hydrochloric acid, filter and wash. Concentrate to about 50 cc., cool, and add sufficient disodium hydrogen phosphate solution to precipitate the magnesium; then add gradually ammonium hydroxid, with constant stirring, until the solution is distinctly alkaline. Determine if the precipitation is complete by the addition of more of the disodium hydrogen phosphate solution. After 30 minutes, add gradually 10 cc. of strong ammonium hydroxid, cover to prevent the escape of ammonia and let stand in the cold. Filter after 12 hours, wash the precipitate free from chlorids, using dilute ammonium hydroxid [**I**, **4** (**d**)], dry, burn, first at a moderate heat, and then ignite intensely and weigh as magnesium pyrophosphate ($Mg_2P_2O_7$).

8 *Method II.—Tentative.*

(Applicable for plant materials other than seeds.)

Calcium.—Make alkaline with ammonium hydroxid the combined filtrates and washings, *C*, under **6**, and proceed as directed under **7** *Calcium*, except that the solution is not to be concentrated. If the ignited calcium oxid has a brown coloration, due to manganese, dissolve in dilute hydrochloric acid and determine the manganese as directed below. Deduct the weight of manganomanganic oxid thus obtained from the weight of the impure calcium oxid.

Manganese.—Acidify the combined filtrates and washings from the calcium determination and evaporate to dryness in a porcelain casserole. Expel the ammonium salts by carefully heating the casserole from above, treat with a few cc. of hydrochloric acid and water, filter off molybdic acid and wash the precipitate until it is free from chlorids. Bring the filtrate to a volume of 100 cc., add 1 or 2 drops of bromin, make alkaline with ammonium hydroxid and let stand for several minutes without agitation. Filter off the precipitated manganese, wash, dry, ignite and weigh the precipitate as manganomanganic oxid. To this add the weight of the manganomanganic oxid found as an impurity in the calcium determination.

Magnesium.—Concentrate the alkaline filtrate from the manganese determination to 75 cc. and determine magnesium as directed under **7**, *Magnesium.*

PHOSPHORIC ACID.

9

Method I.—Official.

Determine phosphoric acid in an aliquot of *A*, under **4**, corresponding to 0.2–1 gram of ash, as directed under **I**, **6** or **9**.

10

Method II.—Official.

Determine phosphoric acid in the plant substance as directed under **I**, **6**, using sufficient material to give 0.2–1 gram of ash in the aliquot of the solution employed.

11

Method III.—Official.

The phosphomolybdate precipitate obtained in **6** is used for the determination of phosphoric acid as directed under **I**, **6**, beginning with "Dissolve the precipitate on the filter with ammonium hydroxid, etc." or as under **I**, **9** (**a**), beginning with "wash with cold water until the filtrate from 2 fillings of the filter yields a pink color, etc."

12

SULPHURIC ACID.—OFFICIAL.

Heat to boiling an aliquot of *A*, under **4**, corresponding to 0.5–1 gram of ash, add barium chlorid solution in small quantities until no further precipitate is formed. Continue the boiling for about 5 minutes and allow to stand for 5 hours or longer in a warm place. Decant the liquid on an ashless filter or tared Gooch, previously heated, treat the precipitate with 15–20 cc. of boiling water, transfer to the filter and wash free from chlorids with boiling water. Dry the precipitate and filter, ignite and weigh as barium sulphate.

SODIUM AND POTASSIUM.

13

Method I.—Official.

(1) To the combined filtrate and washings from the sulphuric acid determination add ammonium hydroxid, drop by drop, until the precipitate formed requires several seconds to dissolve, thus leaving the solution but faintly acid. Heat nearly to the boiling point, and add ammonium hydroxid to precipitate all of the iron, aluminium, etc. Boil in a covered beaker for about 1 minute, remove, and if no ammonia is given off (detected by smelling) continue the addition, drop by drop, until ammonia can be detected. Do not allow the precipitate to settle, but stir and pour on the filter. Wash immediately with hot water, using a fine jet which is played around the edge of the precipitate, thus cutting it free from the paper in order to produce rapid filtration. Wash the precipitate several times, return it to the original beaker, dissolve with a few drops of hydrochloric acid and warm. Reprecipitate the iron, aluminium and phosphoric acid with ammonium hydroxid as directed above, filter and wash until free from chlorids. Evaporate the combined filtrates and washings to dryness, heat below redness until ammonium salts are expelled, dissolve in hot water, add 5 cc. of barium hydroxid solution and heat to boiling; let settle for a few minutes and determine if the precipitation is complete by the addition of barium hydroxid solution to a little

of the clear liquid. When no further precipitate is produced, filter and wash thoroughly with hot water. Heat the filtrate to boiling, add ammonium hydroxid and ammonium carbonate to complete the precipitation of the barium, calcium, etc., let stand a short time on the water bath, filter and wash the precipitate thoroughly with hot water; evaporate the filtrate and washings to dryness, expel ammonium salts by heating below redness, treat with a little hot water, add a few drops of ammonium hydroxid, 1 or 2 drops of ammonium carbonate and a few drops of ammonium oxalate; let stand a few minutes on the water bath, set aside for a few hours, filter, evaporate to complete dryness on the water bath and heat to dull redness until all ammonium salts are expelled and the residue is nearly or quite white. Dissolve in a minimum amount of water, filter into a tared platinum dish, add a few drops of hydrochloric acid, evaporate to dryness on the water bath, heat to dull redness, cool in a desiccator and weigh as potassium and sodium chlorids. Repeat the heating until constant weight is obtained. Dissolve in a small amount of water; if any residue remains, the separation must be repeated until the residue of potassium and sodium chlorids is entirely soluble. Dissolve the residue with water, add an excess of platinum solution [**I**, **40** (**b**)], proceed as directed under **I**, **45**; or, (2) Instead of the foregoing, evaporate to dryness a fresh aliquot of *A*, under **4**, redissolve in water, treat directly with barium hydroxid solution and from this point proceed as directed above in (1).

14 *Method II.—Official.*

Proceed as in **13** through "let stand a short time on the water bath" (the point at which the barium, calcium, etc., have been precipitated with ammonium hydroxid and ammonium carbonate) and then proceed as follows:

Filter into a beaker, add 1 or 2 drops of hydrochloric acid and 1 cc. of ammonium sulphate (75 grams to 1 liter), digest several hours on a water bath, and filter into a tared platinum dish. Evaporate to dryness, heat to full redness, add 1 gram of powdered ammonium carbonate; heat to expel excess of ammonium carbonate, cool and weigh the sulphates of sodium and potassium. Determine potassium as directed under **I**, **42** (**a**).

CHLORIN.

15 *Gravimetric Method.—Official.*

Dissolve a weighed portion of the ash, prepared under **2**, in dilute nitric acid (1 to 10), filter, wash with hot water and determine chlorin in the combined filtrate and washings as directed under **I**, **16** (**a**).

Volumetric Method[1]*.—Official.*

16 REAGENTS.

(**a**) *N/10 silver nitrate.*

(**b**) *N/10 ammonium or potassium thiocyanate.*

(**c**) *Ferric indicator.*—Saturated solution of ferric alum.

(**d**) *Nitric acid.*—Free from lower oxids of nitrogen, secured by diluting the usual pure acid with about ¼ part of water, and boiling till perfectly colorless.

17 DETERMINATION.

Dissolve a weighed portion of the ash, prepared under **2**, in dilute nitric acid (1 to 10), filter and wash with water. Add a known volume of the N/10 silver nitrate in slight excess to the combined filtrate and washings. Stir well, filter and wash the silver chlorid precipitate thoroughly. To the filtrate and washings add 5 cc. of the ferric indicator and a few cc. of the nitric acid. Titrate the excess of silver with the N/10 thiocyanate until a permanent light brown color appears. Calculate the amount of chlorin.

18 POTASSIUM IN PLANTS.—OFFICIAL.

Determine potassium as directed under **I, 41** (**c**), using sufficient plant material to yield 0.5–1 gram of ash in the aliquot of the solution used for the potassium determination.

19 SULPHUR IN PLANTS.—OFFICIAL.

Place 1.5–2.5 grams of material in a nickel crucible of about 100 cc. capacity and add 5 grams of pure anhydrous sodium carbonate. Mix thoroughly, using a nickel or platinum rod, and moisten with approximately 2 cc. of water. Add pure sodium peroxid, approximately 0.5 gram at a time, thoroughly mixing the charge after each addition. Continue adding the peroxid until the mixture becomes nearly dry and quite granular, requiring usually about 5 grams of peroxid. Place the crucible over a low alcohol or other sulphur-free flame and heat carefully with occasional stirring until the contents are fused. (Should the material ignite, the determination is worthless.) After fusion remove the crucible, allow to cool somewhat and cover the hardened mass with peroxid to a depth of about 0.5 cm. Heat gradually, and finally with full flame until fusion again takes place, rotating the crucible from time to time in order to bring any particles adhering to the sides into contact with the oxidizing material. Continue the heating for 10 minutes after fusion is complete. Cool somewhat, place the warm crucible and contents in a 600 cc. beaker and carefully add about 100 cc. of water. After the initial violent action has ceased, wash the material out of the crucible, make slightly acid with hydrochloric acid (adding small portions at a time), transfer to a 500 cc. flask, cool and make to volume. Filter, and determine sulphates in 200 cc. of the filtrate as directed under **12**.

20 CHLORIN IN PLANTS.—OFFICIAL.

Moisten 5 grams of the substance in a platinum dish with 20 cc. of a 5 per cent solution of sodium carbonate, evaporate to dryness and ignite as thoroughly as possible at a temperature not exceeding dull redness. Extract with hot water, filter and wash. Return the residue to the platinum dish and ignite to an ash; dissolve in nitric acid, add this solution to the water extract and determine chlorin as directed under **17**.

BIBLIOGRAPHY.

[1] Sutton. Systematic Handbook of Volumetric Analysis. 10th ed., 1911, p. 145.

III. WATERS.

POTABLE WATER.

TURBIDITY.—OFFICIAL.

1 **REAGENTS.**

(a) *Standard turbidity solution.*—Weigh out 1 gram of elutriated fuller's earth previously dried and sifted through a 200 mesh sieve. Make up to 1 liter with water. If the fuller's earth is of good quality and the proper degree of fineness, this stock solution has a turbidity of 1000. Check the stock solution with a Jackson turbidimeter.

(b) *Turbidity standards.*—Prepared by dilution of (a).

2 **DETERMINATION.**

Determine the turbidity of the sample with a Jackson turbidimeter equipped with either candle or electric light. If the turbidity is less than 100, which prohibits the use of the turbidimeter, determine by direct comparison with turbidity standards contained in bottles of clear white glass.

COLOR.—OFFICIAL.

3 **REAGENTS.**

(a) *Standard color solution.*—Dissolve 1.246 grams of potassium platinic chlorid ($PtCl_4 2KCl$), containing 0.5 gram of platinum, and 1 gram of crystallized cobalt chlorid ($CoCl_2 6H_2O$), containing 0.25 gram of cobalt in a small quantity of water, add 100 cc. of concentrated hydrochloric acid and make up to 1 liter with water. This stock solution has a color of 500.

(b) *Color standards.*—Prepared by dilution of (a).

4 **DETERMINATION.**

Compare the color of the sample, freed from suspended matter, with color standards in tubes of clear white glass.

5 ODOR.—OFFICIAL.

Shake the vessel containing the sample and note the odor. Heat a portion of the sample just to boiling and note the odor.

6 TOTAL SOLIDS.—OFFICIAL.

Thoroughly shake the vessel containing the sample and pipette 100 cc. of the unfiltered water into a weighed platinum dish. If the sample contains much suspended matter, shake, pour rapidly into a 100 cc. measuring cylinder and transfer without delay to a weighed platinum dish; evaporate to dryness and heat to constant weight at 105°C. In the case of highly mineralized waters it is advisable to weigh again after drying at 180°C.

7 SOLIDS IN SOLUTION.—OFFICIAL.

Allow the sample to stand until all sediment has settled and filter if necessary to secure a perfectly clear liquid. Occasionally a clear filtrate can be obtained only by the use of alumina cream but this should be avoided if possible. Evaporate 100–

250 cc. to dryness in a weighed platinum dish. Heat to constant weight at 105°C. In the case of highly mineralized waters it is advisable to weigh again after drying at 180°C.

8 SUSPENDED MATTER.—OFFICIAL.

(1) The difference between the values for total solids and solids in solution represents the suspended matter; or, (2) determine the suspended matter directly by filtering a suitable quantity of the water through a tared Gooch crucible, suitably prepared, and weighing after drying at 105°C.

9 IGNITED RESIDUE.—OFFICIAL.

Ignite the residue from **6** at a low red heat until the ash is white or nearly so. Note any odor or change in color produced during ignition. Record the weight of the ignited residue and calculate the loss on ignition.

FREE AND ALBUMINOID AMMONIA.—OFFICIAL.

10 REAGENTS.

(**a**) *Saturated solution of sodium carbonate.*

(**b**) *Ammonia-free water.*

(**c**) *Standard ammonium chlorid solution.*—One cc. is equivalent to 0.01 mg. of nitrogen in the form of ammonia (NH_3).

(**d**) *Nessler reagent.*—Dissolve 50 grams of potassium iodid in a minimum quantity of cold water. Add a saturated solution of mercuric chlorid until a slight permanent precipitate is formed. Add 400 cc. of a solution containing 200 grams of potassium hydroxid (or an equivalent quantity of sodium hydroxid), dilute to 1 liter, allow to settle and decant.

(**e**) *Alkaline potassium permanganate solution.*—Dissolve 200 grams of potassium hydroxid and 8 grams of potassium permanganate in water and dilute to 1 liter.

11 DETERMINATION.

Connect a flask of about 1500 cc. capacity with an upright bulb condenser by means of a rather large glass tube and a soft rubber stopper or a recently extracted cork stopper. Place in the flask 5 cc. of the saturated solution of sodium carbonate and 500 cc. of ammonia-free water. Distil into 50 cc. Nessler tubes until no further traces of ammonia are indicated on the addition of 2 cc. of the Nessler reagent to 50 cc. of the distillate. Continue the distillation until the volume of the solution in the flask has been reduced to about 200 cc. Cool slightly, add 500 cc. of the water under examination, and distil, at the rate of 1 tubeful in 15 minutes, into 50 cc. Nessler tubes until ammonia ceases to be given off (4 or 5 tubes are usually sufficient). Add 2 cc. of the Nessler reagent to each tube and let stand 10 minutes. Freshly prepare in a similar manner other tubes containing known amounts of the standard ammonium chlorid, made up to 50 cc. with ammonia-free water and compare the nesslerized distillates with these. Report as milligrams per liter of nitrogen in the form of free ammonia (NH_3). Cool the flask and add 50 cc. of the alkaline permanganate recently boiled. Distil, at the rate of 1 tubeful in 15 minutes, into 50 cc. Nessler tubes until ammonia ceases to come off. Nesslerize and compare as in the determination of free ammonia. Report as milligrams per liter of nitrogen in the form of albuminoid ammonia (NH_3).

NITROGEN IN THE FORM OF NITRITE.—OFFICIAL.

12 REAGENTS.

(**a**) *Concentrated hydrochloric acid.*—Sp. gr. 1.2.

(**b**) *Sulphanilic acid solution.*—Dissolve 1 gram of sulphanilic acid in 100 cc. of hot water.

(**c**) *Alpha-naphthylamin hydrochlorid solution.*—Boil 0.5 gram of the salt with 100 cc. of water for 10 minutes at constant volume.

(**d**) *Standard nitrite solution.*—Dissolve 1.1 grams of silver nitrite in nitrite-free water, precipitate the silver with sodium chlorid solution and dilute to 1 liter, mix and allow to settle. Dilute 100 cc. to 1 liter and then 10 cc. of this solution to 1 liter, using in each case nitrite-free water. Each cc. of the last solution is equivalent to 0.0001 mg. of nitrogen as nitrite.

13 DETERMINATION.

Place 100 cc. of the water in a 100 cc. Nessler tube and treat with 1 or 2 drops of concentrated hydrochloric acid. Add 1 cc. of the sulphanilic acid, 1 cc. of the alpha-naphthylamin hydrochlorid, and thoroughly mix. Set aside for 30 minutes with other Nessler tubes containing known amounts of the standard nitrite made up to 100 cc. with nitrite-free water, and treated with hydrochloric acid, sulphanilic acid and alpha-naphthylamin hydrochlorid in the manner just described. Determine the amount of nitrite by comparing the depth of pink color in the known and unknown solutions. Record as nitrogen in the form of nitrite.

NITROGEN IN THE FORM OF NITRATE.

Phenoldisulphonic Acid Method.—Official.

(For water of low chlorin content.)

14 REAGENTS.

(**a**) *Phenoldisulphonic acid solution.*—Dissolve 25 grams of pure white phenol in 150 cc. of concentrated sulphuric acid, add 75 cc. of fuming sulphuric acid (13–15% SO_3) and heat at 100°C. for 2 hours.

(**b**) *Standard nitrate solution.*—Dissolve 0.722 gram of pure potassium nitrate in 1 liter of nitrate-free water. Evaporate 50 cc. of this solution to dryness in a porcelain dish; treat with 2 cc. of the phenoldisulphonic acid solution, rubbing with a glass rod to insure intimate contact. Dilute to 500 cc.; 1 cc. is equivalent to 0.01 mg. of nitrogen as nitrate. This solution is permanent. Standards for comparison are prepared by adding ammonium hydroxid to measured volumes of it in 100 cc. Nessler tubes.

(**c**) *Standard silver sulphate solution.*—Dissolve 4.3969 grams of silver sulphate, free from nitrate, in 1 liter of water; 1 cc. is equivalent to 1 mg. of chlorin.

(**d**) *Ammonium hydroxid.*—Sp. gr. 0.90.

15 DETERMINATION.

Take 100 cc. of the sample, or an amount that will contain 0.05 mg. or less of nitrogen as nitrate, and add standard silver sulphate, precipitating all but about 0.5 mg. of the chlorin. Heat to boiling, allow to settle, or add a little alumina cream, filter and wash with small amounts of hot water. Evaporate the filtrate to dryness in a porcelain dish on the water bath; when cool, treat with 2 cc. of the phenoldisulphonic acid solution as in **14** (**b**). Dilute with water and add slowly ammonium hydroxid until the maximum color is developed. Transfer to a colorimetric cylinder, filter if necessary, and compare with the standards in the usual manner. Record as nitrogen in the form of nitrate.

Reduction Method.—Official.

(For water of high chlorin content.)

16 REAGENTS.

(**a**) *Aluminium foil.*—This reagent should be the purest obtainable. Cut into strips about 10 cm. long, weighing about 0.5 gram.

(**b**) *Sodium or potassium hydroxid solution.*—Dissolve 250 grams of the purest hydroxid obtainable in 1250 cc. of water. Add 2 or 3 strips of aluminium foil, (**a**), and let stand about 12 hours. Concentrate the solution to 1 liter by boiling.

17 DETERMINATION.

Place 100 cc. of the sample, or such a quantity as contains 0.1 mg. or less of nitrogen in the form of nitrate, in a 300 cc. casserole. Add 2 cc. of the sodium hydroxid solution and concentrate by boiling to about one-third original volume. Transfer to a 100 cc. test tube, using nitrogen-free water, diluting if necessary to a volume of about 75 cc. Prepare a blank (preferably several blanks, since the nitrogen impurity in aluminium is often distributed unevenly) by placing about 75 cc. of nitrogen-free water and 2 cc. of the sodium hydroxid solution in a 100 cc. test tube. Place a strip of aluminium foil in each tube. Close the mouths of the test tubes with rubber stoppers connected, by means of bent glass tubes, with other test tubes containing about 50 cc. of slightly acidified ammonia-free water. These latter tubes serve as traps to prevent the escape of ammonia and at the same time permit the free evolution of hydrogen. Allow the sample and blank to stand at room temperature for 12 hours or until reduction is complete. Nesslerize the traps. If high in ammonia, indicating frothing over of the sample, the determination should be discarded. If the traps contain the equivalent of only 1–2 cc. standard ammonia solution each, they should be disregarded. Transfer the sample and blank to distillation flasks, using 250 cc. of ammonia-free water for each, distil, nesslerize and compare with standards as in the determination of free ammonia, **11**. Subtract the quantity of nitrogen found in the blank from that found in the sample. Calculate to milligrams per liter of nitrogen in the form of nitrate.

CHLORIN.—OFFICIAL.

18 REAGENTS.

(**a**) *N/20 sulphuric acid.*

(**b**) *N/20 sodium carbonate.*

(**c**) *Potassium chromate indicator.*—Dissolve 5 grams of potassium chromate in water, add a solution of silver nitrate until a slight permanent red precipitate is produced, filter, and dilute to 100 cc.

(**d**) *Standard silver nitrate solution.*—Dissolve 4.791 grams of silver nitrate in water and dilute to 1 liter; 1 cc. is equivalent to 1 mg. of chlorin. Check by titration against a standardized solution of sodium chlorid.

19 DETERMINATION.

To 100 cc. of the water add a few drops of phenolphthalein. If a red color appears, titrate the carbonates thus indicated to bicarbonates with N/10 or N/20 sulphuric acid. If the water is acid to methyl orange, add N/20 sodium carbonate to neutralize the acidity. Add 1 cc. of the potassium chromate and titrate with the standard silver nitrate. Correct for the amount of silver nitrate necessary to give in 100 cc. of chlorin-free water with 1 cc. of the chromate, the shade obtained at the end of the titration of the sample. Iodids and bromids are not usually found in interfering quantities in potable water. However, if they are present make the equivalent correction.

If chlorids are present in very small quantities, concentrate 500 or 1000 cc. in a porcelain dish to 100 cc., rub down the sides of the dish carefully, add 1 cc. of the indicator and titrate as described above. If sufficient chlorids are present in 100 cc. of the water to consume more than 25 cc. of the standard silver nitrate, determine by precipitation in nitric acid solution and weigh the silver chlorid as directed under **I, 16** (**a**).

OXYGEN REQUIRED.

Method I.—Official.

20

REAGENTS.

(**a**) *Standard potassium permanganate solution.*—Dissolve 0.3952 gram of potassium permanganate in 1 liter of water; each cc. has 0.1 mg. of oxygen available for oxidation.

(**b**) *Standard oxalic acid solution.*—Dissolve 0.7875 gram of crystallized oxalic acid in 1 liter of water.

Determine the value of the oxalic acid in terms of the permanganate by boiling 10 cc. of the oxalic acid and 200 cc. of redistilled water (prepared by treating distilled water with alkaline permanganate and distilling) with 10 cc. of sulphuric acid (1 to 3) and titrating, while still boiling, with the standard permanganate to the appearance of a pink color.

21

DETERMINATION.

Add 10 cc. of sulphuric acid (1 to 3) to 200 cc. of the water in a porcelain dish and heat to boiling. Add from a burette the standard permanganate until the water is distinctly red and boil for 10 minutes, adding more of the standard permanganate from time to time to maintain the red color. Add 10 cc. of the standard oxalic acid and titrate back with the standard permanganate to a pink color. From the total number of cc. of permanganate used, subtract the number of cc. equivalent to 10 cc. of the oxalic acid. The result gives the number of cc. of the permanganate required for 200 cc. of the water. Correct for sulphids, nitrites and ferrous salts, if present, by subtracting the number of cc. of the standard permanganate absorbed by another 200 cc. portion of the sample when treated as above, except that the digestion shall be at room temperature and for a period of 3 minutes.

Method II[1]*.—Official.*

(To be used when the chlorin content of the sample is high.)

22

REAGENTS.

(**a**) *Sodium hydroxid solution.*—Dissolve 50 grams of sodium hydroxid in water, cool and make to 100 cc.

Other reagents and standard solutions are described under **20**.

23

DETERMINATION.

Introduce 100 cc. of the water to be examined in a 300 cc. flask, add 0.5 cc. of the sodium hydroxid and 10 cc. of the permanganate, boil for 10 minutes, allow to cool to 50°-60°C. and add 5 cc. of the dilute sulphuric acid and 10 cc. of the standard oxalic acid. As soon as the liquid has become perfectly colorless, and while constantly agitating, cautiously add from a burette, drop by drop, the standard permanganate, until the liquid acquires a faint permanent redness. The permanganate required to effect this is the quantity required for the decomposition of the organic matter in the 100 cc. of water.

acid and water, filter off molybdic acid and wash the precipitate until it is free from chlorids. Bring the filtrate to a volume of 100 cc., add 1 or 2 drops of bromin, make alkaline with ammonium hydroxid and let stand for several minutes without agitation. Filter off the precipitated manganese, wash, dry, ignite and weigh the precipitate as manganomanganic oxid. To this add the weight of the manganomanganic oxid found as an impurity in the calcium determination.

Magnesium.—Concentrate the alkaline filtrate from the manganese determination to 75 cc. and determine magnesium as directed under **7**, *Magnesium.*

PHOSPHORIC ACID.

9 *Method I.—Official.*

Determine phosphoric acid in an aliquot of *A*, under **4**, corresponding to 0.2–1 gram of ash, as directed under **I**, **6** or **9**.

10 *Method II.—Official.*

Determine phosphoric acid in the plant substance as directed under **I**, **6**, using sufficient material to give 0.2–1 gram of ash in the aliquot of the solution employed.

11 *Method III.—Official.*

The phosphomolybdate precipitate obtained in **6** is used for the determination of phosphoric acid as directed under **I**, **6**, beginning with "Dissolve the precipitate on the filter with ammonium hydroxid, etc." or as under **I**, **9** (**a**), beginning with "wash with cold water until the filtrate from 2 fillings of the filter yields a pink color, etc."

12 SULPHURIC ACID.—OFFICIAL.

Heat to boiling an aliquot of *A*, under **4**, corresponding to 0.5–1 gram of ash, add barium chlorid solution in small quantities until no further precipitate is formed. Continue the boiling for about 5 minutes and allow to stand for 5 hours or longer in a warm place. Decant the liquid on an ashless filter or tared Gooch, previously heated, treat the precipitate with 15–20 cc. of boiling water, transfer to the filter and wash free from chlorids with boiling water. Dry the precipitate and filter, ignite and weigh as barium sulphate.

SODIUM AND POTASSIUM.

13 *Method I.—Official.*

(1) To the combined filtrate and washings from the sulphuric acid determination add ammonium hydroxid, drop by drop, until the precipitate formed requires several seconds to dissolve, thus leaving the solution but faintly acid. Heat nearly to the boiling point, and add ammonium hydroxid to precipitate all of the iron, aluminium, etc. Boil in a covered beaker for about 1 minute, remove, and if no ammonia is given off (detected by smelling) continue the addition, drop by drop, until ammonia can be detected. Do not allow the precipitate to settle, but stir and pour on the filter. Wash immediately with hot water, using a fine jet which is played around the edge of the precipitate, thus cutting it free from the paper in order to produce rapid filtration. Wash the precipitate several times, return it to the original beaker, dissolve with a few drops of hydrochloric acid and warm. Reprecipitate the iron, aluminium and phosphoric acid with ammonium hydroxid as directed above, filter and wash until free from chlorids. Evaporate the combined filtrates and washings to dryness, heat below redness until ammonium salts are expelled, dissolve in hot water, add 5 cc. of barium hydroxid solution and heat to boiling; let settle for a few minutes and determine if the precipitation is complete by the addition of barium hydroxid solution to a little

of the clear liquid. When no further precipitate is produced, filter and wash thoroughly with hot water. Heat the filtrate to boiling, add ammonium hydroxid and ammonium carbonate to complete the precipitation of the barium, calcium, etc., let stand a short time on the water bath, filter and wash the precipitate thoroughly with hot water; evaporate the filtrate and washings to dryness, expel ammonium salts by heating below redness, treat with a little hot water, add a few drops of ammonium hydroxid, 1 or 2 drops of ammonium carbonate and a few drops of ammonium oxalate; let stand a few minutes on the water bath, set aside for a few hours, filter, evaporate to complete dryness on the water bath and heat to dull redness until all ammonium salts are expelled and the residue is nearly or quite white. Dissolve in a minimum amount of water, filter into a tared platinum dish, add a few drops of hydrochloric acid, evaporate to dryness on the water bath, heat to dull redness, cool in a desiccator and weigh as potassium and sodium chlorids. Repeat the heating until constant weight is obtained. Dissolve in a small amount of water; if any residue remains, the separation must be repeated until the residue of potassium and sodium chlorids is entirely soluble. Dissolve the residue with water, add an excess of platinum solution [**I**, **40** (**b**)], proceed as directed under **I**, **45**; or, (2) Instead of the foregoing, evaporate to dryness a fresh aliquot of *A*, under **4**, redissolve in water, treat directly with barium hydroxid solution and from this point proceed as directed above in (1).

14 *Method II.—Official.*

Proceed as in **13** through "let stand a short time on the water bath" (the point at which the barium, calcium, etc., have been precipitated with ammonium hydroxid and ammonium carbonate) and then proceed as follows:

Filter into a beaker, add 1 or 2 drops of hydrochloric acid and 1 cc. of ammonium sulphate (75 grams to 1 liter), digest several hours on a water bath, and filter into a tared platinum dish. Evaporate to dryness, heat to full redness, add 1 gram of powdered ammonium carbonate; heat to expel excess of ammonium carbonate, cool and weigh the sulphates of sodium and potassium. Determine potassium as directed under **I**, **42** (**a**).

CHLORIN.

15 *Gravimetric Method.—Official.*

Dissolve a weighed portion of the ash, prepared under **2**, in dilute nitric acid (1 to 10), filter, wash with hot water and determine chlorin in the combined filtrate and washings as directed under **I**, **16** (**a**).

Volumetric Method[1]*.—Official.*

16 REAGENTS.

(**a**) *N/10 silver nitrate.*

(**b**) *N/10 ammonium or potassium thiocyanate.*

(**c**) *Ferric indicator.*—Saturated solution of ferric alum.

(**d**) *Nitric acid.*—Free from lower oxids of nitrogen, secured by diluting the usual pure acid with about ¼ part of water, and boiling till perfectly colorless.

17 DETERMINATION.

Dissolve a weighed portion of the ash, prepared under **2**, in dilute nitric acid (1 to 10), filter and wash with water. Add a known volume of the N/10 silver nitrate in slight excess to the combined filtrate and washings. Stir well, filter and wash the silver chlorid precipitate thoroughly. To the filtrate and washings add 5 cc. of the acid to clear the solution. Heat to boiling and neutralize with ammonium hydroxid, stirring constantly. Add ammonium hydroxid in slight excess and allow to stand 3 hours in a warm place. Filter off the supernatant liquid and wash the precipitate once or twice by decantation with 1 per cent ammonium oxalate solution. Dissolve the precipitate in hydrochloric acid, dilute to 100–200 cc., add a little oxalic acid and precipitate as above. After standing 3 hours, filter, wash with the ammonium oxalate solution as above, dry, ignite, heat over a blast lamp and weigh as calcium and strontium oxids. Subtract from this weight, the weight of strontium oxid equivalent to the strontium under **46**. The difference is the weight of calcium oxid. Calculate to calcium. Designate the combined filtrates and washings as *C*.

As a check on the calcium oxid, evaporate to dryness the filtrate from the strontium nitrate under **46**, beginning with "Filter, wash with ether-alcohol mixture, etc.," dissolve the calcium nitrate in water, precipitate as oxalate, filter, wash, ignite and weigh as calcium oxid.

46 STRONTIUM.—TENTATIVE.

Dissolve the oxids under **45** in dilute nitric acid and test with the spectroscope for strontium. If strontium is present, transfer the nitric acid solution to a small Erlenmeyer flask. Evaporate nearly to dryness over a low flame and heat in an air bath at 150°–160°C. for 1–2 hours after the water is evaporated. Break up the dried material with a stirring rod, add 10–15 cc. of a mixture of equal parts of absolute alcohol and ether to dissolve the calcium nitrate. Cork the flask and allow to stand with frequent shaking for 2 hours or longer. Decant the solution through a 5.5 cm. filter, preserving the filtrate. Wash the residue several times by decantation with small portions of ether-alcohol solution. Dry the residue and the filter paper and wash the filter paper repeatedly with small portions of hot water, collecting the filtrate in the flask containing the main portion of the strontium nitrate residue. Add 1 or 2 drops of dilute nitric acid, evaporate, dry, pulverize and treat with 10–15 cc. of ether-alcohol mixture as above. Cork the flask and let stand about 12 hours with occasional shaking. Filter, wash with ether-alcohol mixture until a few drops of the filtrate evaporated on a watch glass leave practically no residue. Dry the paper and precipitate. Dissolve the strontium nitrate in a few cc. of hot water. Add a few drops of sulphuric acid, then a volume of alcohol equal to the volume of the solution and allow to stand 12 hours. Filter, ignite, weigh as strontium sulphate and calculate to strontium. Test spectroscopically for calcium and barium. If these elements are present, determine the amount and make the necessary correction.

47 MAGNESIUM.—OFFICIAL.

Concentrate *C*, under **45**, to about 200 cc.; add 2–3 grams of diammonium hydrogen phosphate and sufficient hydrochloric acid to clear the solution when the ammonium phosphate is all dissolved; disodium hydrogen phosphate or sodium ammonium hydrogen phosphate may be used instead of the diammonium hydrogen phosphate. When cold, make slightly alkaline with ammonium hydroxid, stirring constantly. Add 1–2 cc. excess of ammonium hydroxid and allow to stand about 12 hours. Filter off the supernatant liquid and wash 3 or 4 times by decantation with a solution of 2.5 per cent ammonium hydroxid. Dissolve the precipitate in hydrochloric acid, dilute to about 150 cc., add a little diammonium hydrogen phosphate and precipitate with ammonium hydroxid as before. Allow to stand 6–12 hours, filter, wash free from chlorids, ignite, heat over a blast lamp and weigh as magnesium pyrophosphate. (Cf. II, **7**.) Calculate to magnesium.

SULPHURIC ACID, SODIUM, POTASSIUM AND LITHIUM.

48 SULPHURIC ACID.—OFFICIAL.

Make a preliminary examination, using 100–250 cc. of the water to determine the approximate quantity of sulphates. The alkali salts present can be approximated by calculating the amount of sodium necessary to combine with the excess of acids (hydrochloric, sulphuric, and bicarbonic) over the calcium and magnesium.

Take a quantity, usually 1–5 liters, of the water sufficient to yield not more than 1 gram of barium sulphate and not more than 0.5 gram of mixed chlorids. Acidify with hydrochloric acid, evaporate to dryness in a platinum dish, and remove silica by two evaporations as under **40**, using not more than 2 cc. of hydrochloric acid (1 to 1) for the final solution. Combine the filtrate and washings from the silica determinations, and concentrate to about 150–200 cc. Heat to boiling and precipitate with slight excess of 10 per cent barium chlorid solution, added very slowly and with constant stirring. Cover and allow to stand on the steam bath about 12 hours. Filter, wash thoroughly the precipitate of barium sulphate with hot water, dry, ignite over a Bunsen burner and weigh.

If the content of sulphate in the sample is unusually large, proceed as far as the concentration of the silica filtrates as directed above. Add 50 cc. of concentrated hydrochloric acid, heat to boiling and precipitate with barium chlorid solution as before. Evaporate to dryness, wash the precipitate repeatedly by decantation and filter. Complete the washing of the precipitate; ignite and weigh. Calculate to the sulphuric acid ion. Designate the filtrate as *E*.

SODIUM, POTASSIUM AND LITHIUM.

Amyl Alcohol Method.—Official.

49 PREPARATION OF THE MIXED CHLORIDS.

Evaporate to dryness *E*, under **48**, in a platinum dish and ignite the residue to faint redness to remove all traces of ammonium salts. Dissolve the residue in the dish in about 200 cc. of water and precipitate with milk of lime or a solution of barium hydroxid. Boil, allow to stand for 30 minutes, and filter off the insoluble magnesium hydroxid and undissolved lime. Thoroughly wash the precipitate with hot water and combine the filtrate and washings. If the precipitate of magnesium is large, it is advisable to dissolve in a small amount of hydrochloric acid, evaporate to dryness, take up with water, and precipitate as before. Concentrate the two filtrates and washings to 200–250 cc. Add ammonium hydroxid and sufficient ammonium carbonate to precipitate the calcium and barium. Allow to stand on a steam bath for 1–2 hours. Filter off the supernatant liquid, dissolve the precipitate in hydrochloric acid, reprecipitate as above, and wash thoroughly with hot water. Evaporate the combined filtrates and washings to dryness and drive off the ammonium salts by gentle heat. Treat the residue with water; filter through a small filter, using as little wash water as possible; evaporate to a small volume and again precipitate with 1 or 2 drops of ammonium hydroxid and 2 or 3 drops of ammonium carbonate and oxalate. If any precipitate appears (which is usually not the case) filter and repeat the process. Evaporate the filtrate to dryness and drive off all ammonium salts by heating in platinum to faint redness. Treat the residue with a little water; filter into a small platinum dish; add a few drops of hydrochloric acid and evaporate to dryness. Dry in an oven, heat to faint redness, cool in a desiccator and weigh the combined chlorids of potassium, sodium and lithium. Repeat the heating to constant weight (x). Dissolve the mixed chlorids in hot water; filter and wash. Return the filter paper and residue to the dish, dry, ignite and weigh (y). The difference between (x) and (y) is the weight of the mixed chlorids.

50 DETERMINATION[4].

Transfer the combined chlorids to a 50–100 cc. Erlenmeyer flask and evaporate the solution nearly, but not quite, to dryness. Add about 30 cc. of redistilled amyl alcohol. Connect the flask, the stopper of which carries a thermometer, with a condenser if desired, to avoid the escape of the irritating vapor of the amyl alcohol, and boil until the temperature rises to the boiling point of amyl alcohol (approximately 130°C.) to remove the water. Cool slightly and add a drop of hydrochloric acid to convert small amounts of lithium hydroxid to lithium chlorid. Connect with the condenser and repeat the boiling until the temperature reaches the boiling point of amyl alcohol to again drive off the water. The contents of the flask at this time are usually 15–20 cc. Filter through a small paper or a Gooch crucible into a graduated cylinder and note the exact quantity of the filtrate, which determines the subsequent correction. Wash the precipitate with small quantities of amyl alcohol. If the quantity of lithium exceeds 2–3 mg., dissolve the precipitate from the flask and filter with hot water, and repeat the separation by boiling again in amyl alcohol. Filter and wash as before, noting the exact quantity of filtrate exclusive of washings. Evaporate the filtrates and washings in a small platinum dish to dryness on the steam bath, dissolve the residue in water and add a few drops of sulphuric acid. Evaporate on a steam bath and expel the excess of sulphuric acid by heating gently over a Bunsen burner until the carbonaceous matter is completely burned off, repeating the addition of a few drops of sulphuric acid if necessary. Cool and weigh the dish and contents (x). Dissolve in a small quantity of hot water, filter through a small filter, wash and return filter to dish; ignite and weigh (y). The difference between (x) and (y) is the weight of impure lithium sulphate.

The purity of the lithium sulphate should be tested by adding small amounts of ammonium phosphate solution and ammonium hydroxid, which will precipitate any magnesium present in the lithium sulphate. Any precipitate appearing after standing overnight should be collected on a small filter, ignited, weighed as magnesium pyrophosphate, calculated to sulphate and subtracted from the weight of the impure lithium sulphate. From this weight subtract 0.00113 gram of sodium and potassium sulphates for every 10 cc. of amyl alcohol filtrates, exclusive of the amyl alcohol used in washing the residue, on account of the solubility of sodium and potassium chlorids in amyl alcohol. Calculate to lithium from the corrected weight of lithium sulphate, using the factor 0.1263.

Dissolve the mixed chlorids from the flask with hot water and filter, evaporate to dryness, ignite gently to remove amyl alcohol, filter and thoroughly wash; concentrate the filtrate and washings to 25–50 cc. Transfer to a porcelain dish, add sufficient platinum solution [I, **40** (**b**)] to convert sodium and potassium to their respective double chlorids and evaporate to dryness. Treat the residue with 80 per cent alcohol, filter and wash until the excess of platinic chlorid and sodium platinic chlorid has been removed. Dry the filter and precipitate, dissolve the residue in hot water, and transfer to a weighed platinum dish. Evaporate on a steam bath, dry for 30 minutes in an oven at 100°C. and weigh as potassium platinic chlorid; calculate to potassium chlorid, using the factor 0.3067. To the weight of potassium chlorid add 0.00051 gram for every 10 cc. of amyl alcohol used in the extraction of the lithium chlorid, which corrects for the solubility of the potassium chlorid in amyl alcohol. Calculate to potassium, using the factor 0.5244.

The weight of sodium chlorid is found by subtracting the combined corrected weights of lithium chlorid and potassium chlorid from the total weight of the 3 chlorids. Calculate the sodium chlorid to sodium, using the factor 0.3934.

51 *Ether-Alcohol Method*[6].—*Official.*

Dissolve the mixed chlorids obtained as directed in **49** in a minimum amount of cold water (about 1.5 cc. will be more than sufficient for 0.5 gram of the salts), introducing the solution into a tall 200 cc. beaker. Add 1 drop of concentrated hydrochloric acid, and then add *gradually* 20 cc. of absolute alcohol, dropping the latter into the center of the beaker (not on the sides) while rotating the solution. The sodium and potassium chlorids should be precipitated in a perfectly uniform granular condition. In a similar manner, while rotating the mixture, add 60 cc. of ether (sp. gr. 0.716–0.717 at 25°C.) and allow the mixture to stand about 5 minutes or until the precipitate is well agglomerated and the supernatant liquid almost clear, rotating the mixture occasionally during this period. Filter through a tared Gooch crucible into an Erlenmeyer flask by means of suction, using a bell jar arrangement, washing the beaker thoroughly with a mixture of 1 part alcohol and 5 parts ether, collecting all of the precipitate on the Gooch with the aid of a rubber tipped rod. After thorough washing of the precipitate on the Gooch set the latter aside and rinse the funnel with alcohol-ether mixture to wash any adhering lithium solution into the flask containing the filtrate. Evaporate the filtrate to dryness on a steam bath, using an air blast. Treat the residue with 10 cc. of *absolute* alcohol, warming if necessary, so that practically all of the residue dissolves. If a slight film remains on the bottom and sides of the flask, remove it with a rubber tipped rod. Then, while rotating the solution in the flask, add 50 cc. of ether (sp. gr. 0.716–0.717 at 25°C.), followed by *1 drop* of concentrated hydrochloric acid. Allow to stand for 30 minutes, rotating the solution at frequent intervals. When the fine precipitate has agglomerated (only a very small amount usually being precipitated), filter into a tall beaker by means of suction through the Gooch crucible containing the first precipitate. Wash the combined precipitates with the ether-alcohol mixture, using the same precautions as in the case of the first precipitation. Dry the Gooch and its contents in an oven, ignite gently, cool and weigh, obtaining in this manner the weight of combined sodium and potassium chlorids. Reserve for the determination of potassium.

Evaporate on a steam bath the ether-alcohol filtrate and washings containing the lithium. Dissolve the residue in a little water, add a slight excess of sulphuric acid and transfer to a weighed porcelain or platinum dish. Evaporate as far as possible on a steam bath and then gently ignite the residue over a flame. By placing the dish on a triangle over an asbestos gauze and using a low flame, the solution can be evaporated without spattering. Finally ignite carefully over a full flame, cool and weigh. When charring has occurred, it is well to repeat the ignition with sulphuric acid. Calculate to lithium, using the factor 0.1263.

Remove the potassium and sodium chlorids from the Gooch crucible by washing with 25–50 cc. of hot water, using suction, and collecting the filtrate in a porcelain dish. Add sufficient platinum solution [**I, 40 (b)**] to convert the potassium and sodium to their respective double chlorids and evaporate to dryness. Treat the residue with 80 per cent alcohol, filter, and wash until the excess of platinic chlorid and sodium platinic chlorid has been removed. Dry the filter and precipitate, dissolve the residue in hot water, and transfer to a weighed platinum dish. Evaporate on a steam bath, dry for 30 minutes in an oven at 100°C., cool, and weigh as potassium platinic chlorid. Calculate to potassium chlorid, using the factor 0.3067; and to potassium, using the factor 0.1609.

Find the weight of sodium chlorid by subtracting the weight of potassium chlorid from the weight of combined potassium and sodium chlorids obtained above. Calculate to sodium, using the factor 0.3934.

BARIUM.—TENTATIVE.

It is not necessary to look for barium if sulphate is present in appreciable amount unless the water contains a large amount of bicarbonate or chlorid, which may hold in solution a small amount of both sulphate and barium.

52 REAGENTS.

(**a**) *Ammonium dichromate solution.*—Dissolve 100 grams of the salt in water and make up to 1 liter.

(**b**) *Ammonium acetate solution.*—Dissolve 300 grams of the salt in water, neutralize with ammonia and make up to 1 liter.

(**c**) *Dilute ammonium acetate solution.*—Dilute 20 cc. of (**b**) to 1 liter.

Reaction of acetate solutions should be alkaline rather than acid.

53 *Gravimetric Method.*

Acidify a 1–5 liter portion of the sample with hydrochloric acid and concentrate to about 200 cc. (if a precipitate forms it should be filtered off and examined for barium). Add about 0.5 gram of ammonium chlorid and precipitate the iron and aluminium with ammonium hydroxid. Boil, filter and wash. To the filtrate add an excess of the ammonium acetate solution, 10 cc., **52** (**b**), keeping the total volume about 200 cc. Heat to boiling and add, with stirring, about 5 cc. of the ammonium dichromate solution. Allow to settle and cool. Decant the clear liquid through a filter, wash the precipitate by decantation with the dilute ammonium acetate solution, **52** (**c**), until the filtrate is no longer perceptibly colored (100 cc. of wash solution). Place the beaker under the funnel, dissolve the precipitate on the paper with warm dilute nitric acid, using as little as possible, and wash the paper. Add a little more acid to dissolve the precipitate in the beaker, then ammonium hydroxid until the precipitate forming again no longer redissolves. Heat to boiling, add, with stirring, 10 cc. of the ammonium acetate solution and 2 cc. of the ammonium dichromate solution, allow to cool slowly and wash the precipitate by decantation with the dilute ammonium acetate solution. Dry the barium chromate, burn the filter separately, ignite moderately to constant weight and weigh as barium chromate ($BaCrO_4$). Record as barium, using the factor 0.54217.

54 *Volumetric Method.*

Proceed as directed under **53** through "wash the precipitate by decantation with the dilute ammonium acetate solution" (after the second precipitation). Then proceed as follows:

Dissolve the precipitate in about 10 cc. of dilute hydrochloric acid (1 to 1) and hot water. Wash the filter, dilute the solution to about 400 cc., and add about 50 cc. of a freshly prepared 10 per cent solution of potassium iodid. Mix carefully and titrate the liberated iodin after 3–4 minutes with standard thiosulphate (1 cc. N/10 thiosulphate = 4.579 mg. barium).

PHOSPHORIC ACID.—OFFICIAL.

55 REAGENTS.

The reagents used are described under **I, 7.**

56 DETERMINATION.

Treat 500 cc. of the water, or a larger amount if necessary, with about 10 cc. of concentrated nitric acid and evaporate in a porcelain dish nearly to dryness to drive off hydrochloric acid. Treat the residue with water, and filter if necessary. Add ammonium hydroxid to alkalinity and then just enough nitric acid to restore acidity.

Add some solid ammonium nitrate and heat in the water bath at a temperature of 45°–50°C. Add the molybdate solution and keep at the above temperature for 30 minutes. The yellow precipitate formed at this point appears generally only in traces; if more than traces are present, filter and wash with cold water until entirely free from nitric and molybdic acids. Transfer the precipitate and filter to a beaker, add a little water and beat the paper and contents to a pulp. Dissolve the yellow precipitate in a small amount of the standard potassium hydroxid; add phenolphthalein and titrate with the standard acid. From the data so obtained calculate the phosphoric acid ion in the water to milligrams per liter.

57 MANGANESE, IODIN, BROMIN, ARSENIC AND BORIC ACID.

Evaporate large quantities of the water to dryness, after the addition of small amounts of solid sodium carbonate. Boil the residue thus obtained with water, transfer to a filter and wash thoroughly with hot water. Use the residue remaining on the filter for the determination of manganese. Make the alkaline filtrate up to a definite volume and use for the determination of iodin, bromin, arsenic and boric acid.

MANGANESE.

Persulphate Method.—Official.

58 REAGENTS.

(**a**) *Dilute nitric acid (1 to 1).*

(**b**) *Silver nitrate solution.*—Dissolve 2 grams of silver nitrate in 1 liter of water.

(**c**) *Ammonium persulphate.*

(**d**) *Standard manganous sulphate solution.*—Dissolve 0.2877 gram of pure potassium permanganate in a small amount of water, add an excess of sulphuric acid, reduce carefully with oxalic acid and make up to 1 liter. One cc. of this solution is equivalent to 0.1 mg. of manganese.

59 DETERMINATION.

Dissolve the insoluble residue under **57** in an excess of the dilute nitric acid, evaporate to dryness, treat with water, add about 1 cc. of strong nitric acid and a little of the silver nitrate. If a precipitate of silver chlorid appears, add more of the silver nitrate until all the chlorin is precipitated. Add an excess of about 10 cc. of the silver nitrate for each mg. of manganese present in the sample. Filter, add 1 gram of ammonium persulphate to the filtrate and place the beaker or flask containing the solution on the steam bath until a pink color develops (usually about 20 minutes). Compare the color developed with standards similarly prepared by treating solutions containing known amounts of the standard manganous sulphate with nitric acid, silver nitrate and ammonium persulphate.

Bismuthate Method.—Tentative.

60 REAGENTS.

(**a**) *Dilute nitric acid (1 to 4).*—Free from brown oxid of nitrogen by aeration.

(**b**) *Sulphuric acid (1 to 3).*

(**c**) *Dilute sulphuric acid.*—Dilute 25 cc. of concentrated acid to 1 liter with water. Add enough permanganate solution to color faintly the dilute acid.

(**d**) *Standard manganous sulphate solution.*—Dissolve 0.2877 gram of pure potassium permanganate in about 100 cc. of water, acidify the solution with sulphuric acid and heat to boiling. Add slowly a sufficient quantity of a dilute solution of oxalic acid to discharge the color. Cool and dilute to 1 liter. One cc. of this solution is equivalent to 0.1 mg. of manganese. The standard should be prepared by following the same procedure as is used for the sample. This solution is more permanent than a solution of potassium permanganate, which may, however, be used. To prepare it, dissolve 0.288 gram of potassium permanganate in water and dilute the solution to 1 liter.

(**e**) *Sodium bismuthate.*—Pure dry salt.

61 DETERMINATION.

Remove chlorin by two or more evaporations with sulphuric acid from such a quantity of the sample as contains 1 mg. or less of manganese. The residue obtained under **57** may be used in lieu of a fresh sample by dissolving it in an excess of dilute nitric acid, adding sulphuric acid and removing chlorin by two or more evaporations. In either case, volatilize the sulphuric acid and ignite the residue gently (less than 500°C.). Dissolve in 40 cc. of nitric acid, add about 0.5 gram of sodium bismuthate and heat until the permanganate color disappears. Add a few drops of a solution of ammonium or sodium bisulphate to clear the solution and again boil to expel oxids of nitrogen. Remove from the source of heat, cool to 20°C., again add 0.5 gram of sodium bismuthate and stir. When the maximum permanganate color has developed, filter through an alundum or Gooch crucible containing an asbestos mat which has been ignited, treated with a solution of potassium permanganate and washed with water. Wash the precipitate with dilute sulphuric acid until the washings are colorless. Transfer the filtrate to a colorimeter tube and compare the color with that of standards prepared from the potassium permanganate solution. To prepare the standards, dilute with sulphuric acid, **60** (**c**), portions of 0.2, 0.4, 0.6 cc., etc., of the permanganate solution to the same volume as the filtrate.

IODIN AND BROMIN.—TENTATIVE.

62 REAGENTS.

(**a**) *Sodium hydroxid solution.*—Dissolve 10 grams of sodium hydroxid in water, cool and dilute to 100 cc.

(**b**) *Sulphuric acid (1 to 5).*

(**c**) *Potassium or sodium nitrite solution.*—Dissolve 2 grams of potassium or sodium nitrite in 100 cc. of water.

(**d**) *Carbon disulphid.*—Freshly purified by distillation.

(**e**) *Chlorin water.*—Saturated and freshly prepared.

63 DETERMINATION.

Evaporate to dryness an aliquot of the alkaline filtrate under **57**, add 2-3 cc. of water to dissolve the residue and enough 95 per cent alcohol to make the percentage of alcohol about 90. This precipitates the chlorids. Heat to boiling, filter and repeat the preceding solution and precipitation once or twice. Add 2 or 3 drops of the sodium hydroxid to the combined alcoholic filtrates and evaporate to dryness. Dissolve this last residue in 2-3 cc. of water and repeat as above described the precipitation with alcohol, heating and filtering. Add a drop of the sodium hydroxid to this alcoholic filtrate and evaporate to dryness. Dissolve this residue in a little water, acidify with the sulphuric acid, using 3 or 4 drops in excess, and transfer to a small flask. Add 4 drops of the potassium nitrite and about 5 cc. of the carbon disulphid. Shake until all the iodin is extracted, filter off the acid solution from the carbon disulphid, retaining the latter in the flask. Wash the flask, filter and contents with cold water and transfer the carbon disulphid, containing the iodin in solution, to a Nessler tube, using approximately 5 cc. of the carbon disulphid. In washing the filter, make the contents of the tube up to definite volume, usually 12-15 cc., and compare the color with that of other tubes containing known amounts of iodin dissolved in carbon disulphid. Prepare these standard tubes by treating measured quantities of a solution of known potassium iodid content as described above, beginning with "acidify with the sulphuric acid". Transfer the sample and standards, from which the iodin has been removed, severally to small flasks. To the standards add definite measured quantities of a bromid solution of known strength, and to each of the flasks containing sample and standards add 5 cc. of purified carbon disulphid. Add the saturated chlorin water, 1 cc. at a time, shaking after each addition until all the bromin is set free. (Avoid a large excess of chlorin, since a bromo-chlorid may be formed which spoils the color reaction.) Filter off the water solution from the carbon disulphid through a moistened filter, wash the contents of the filter 2 or 3 times with water and then transfer to a Nessler tube by means of about 1 cc. of carbon disulphid. Repeat this extraction of the filtrate twice, using 3 cc. of carbon disulphid each time. The combined carbon disulphid extracts usually amount to 11.5–12 cc. Add enough carbon disulphid to the tubes to bring them to a definite volume, usually 12–15 cc., and compare the sample with the standards. In some cases, when using this method near its upper limit the amounts of carbon disulphid recommended do not extract all the bromin. In these cases, make one or two extra extractions with carbon disulphid, transfer the extracts to another tube and compare the color with some of the lower standards and add the readings thus obtained to the others.

Results closely approximating the true values for iodin and bromin can be obtained on most samples by omitting the extractions with alcohol given above and by comparing the color of the carbon disulphid solutions directly in the extraction flasks, thus shortening the method.

ARSENIC.

Method I.—Official.

64 REAGENTS.

(a) *Zinc, arsenic-free.*

(b) *Sulphuric acid (1 to 5), arsenic-free.*

(c) *Standard arsenious oxid solution.*—Dissolve 0.0132 gram of pure arsenious oxid in 100 cc. of water containing about 50 mg. of sodium carbonate. One cc. of this solution is equivalent to 0.1 mg. of As.

65 DETERMINATION.

Evaporate to dryness an aliquot of the alkaline filtrate under **57**. Acidify with the sulphuric acid and subject to the action of the zinc and the sulphuric acid in a Marsh-Berzelius apparatus. Compare the mirror obtained with a mirror prepared from an arsenious oxid solution of known strength. Calculate to the arsenic acid ion.

Method II.—Tentative.

66 REAGENTS AND APPARATUS.

Described under **XI**, **1** and **2** (Fig. 5).

67 DETERMINATION.

Take such a portion of the alkaline filtrate under **57** as contains not more than 0.03 mg. of arsenious oxid (As_2O_3). If the quantity taken is greater than 10 cc., evaporate the solution to about that volume on the water bath. Transfer the solution into the generator of the apparatus described under **XI**, **2**, with the aid of about 10 cc. of water; add 20 cc. of dilute sulphuric acid (1 to 2) and proceed as directed under **XI**, **4**, beginning with "If the total volume is less than 40 cc., dilute to that volume with water and add 4 cc. of the 20 per cent potassium iodid solution".

68 BORIC ACID.—OFFICIAL.

(Glassware containing boron must not be used in this determination.)

Qualitative test.—Evaporate to dryness a part of the alkaline filtrate under **57**, treat with 1-2 cc. of water and slightly acidify with dilute hydrochloric acid (1 to 1). Add about 25 cc. of 95 per cent alcohol, boil, filter and repeat the extraction of the residue. Make the filtrate slightly alkaline with sodium hydroxid solution and evaporate to dryness. Add a little water, slightly acidify with dilute hydrochloric acid and place a strip of turmeric paper in the liquid. Evaporate to dryness on the steam bath and continue the heating until the turmeric paper is dry. If boric acid is present, the turmeric paper takes on a cherry-red color. As a confirmatory test, apply a drop of dilute ammonium hydroxid to the reddened paper, and a dark olive color will be due to boric acid.

Quantitative test.—It is not usually necessary to determine boric acid quantitatively. However, if it is necessary, the Gooch method[7] is used.

69 METHOD OF REPORTING RESULTS.—TENTATIVE.

Report the bases and acids as positive and negative ions in milligrams per liter, except in the case of silica, which report as such without considering how much is present as the silicic acid ion and how much as free silica. Report iron and aluminium together when present in unimportant quantities, and in calculations consider them as iron. When iron and aluminium are present in larger quantities, make the separation and report each separately.

In calculating the hypothetical combinations of acid and basic ions, join sodium to nitrous, nitric, metaboric and arsenic acids; potassium to iodin and bromin; calcium to phosphoric acid. Assign the residual basic ions in the following order: Ammonium, lithium, potassium, sodium, magnesium, calcium, strontium, manganese, iron and aluminium—to the residual acid ions in the following order: Chlorin, sulphuric acid ion, carbonic acid ion, and bicarbonic acid ion. In case the bicarbonic acid ion is not present in a sufficient quantity to join with all the calcium, the residual calcium is joined to silica to form calcium silicate, and manganese, iron and aluminium are calculated to the oxids Mn_3O_4, Fe_2O_3 and Al_2O_3, respectively.

INDUSTRIAL WATER.

70 SOLIDS IN SOLUTION.—OFFICIAL.

Determine as directed under **7**.

71 CHLORIN.—OFFICIAL.

Determine as directed under **19**.

72 COMBINED CARBONIC AND BICARBONIC ACIDS.—OFFICIAL.

Determine as directed under **39**.

73 NITRATES.—OFFICIAL.

Determine as directed under **15** or **17**.

74 SILICA.—OFFICIAL.

Determine as directed under **40**. Generally one evaporation with hydrochloric acid for removal of silica is sufficient.

75 IRON AND ALUMINIUM.—OFFICIAL.

Determine as directed under **41**.

76 CALCIUM.—OFFICIAL.

If no phosphoric acid is present, concentrate the filtrate from the determination of iron and precipitate with ammonium hydroxid and oxalate as directed under **45**. Usually one precipitation is sufficient.

77 MAGNESIUM.—OFFICIAL.

Determine as directed under **47**.

78 SULPHURIC ACID AND ALKALIES.—OFFICIAL.

Follow the methods prescribed under **48** and **50** or **51**. Generally, however, for technical purposes it is sufficiently accurate to determine the acids and the bases, except sodium and potassium, and then to calculate the excess of acid over basic ions to the sodium salt, and state the alkali thus found as sodium and potassium by difference.

79 TEMPORARY HARDNESS.—OFFICIAL.

The difference between the alkalinity after boiling, **83**, and the alkalinity before boiling, **81**, is the temporary hardness in parts per million of calcium carbonate.

ALKALINITY[8].—Before Boiling.—Official.

80 REAGENTS.

(**a**) *N/50 sulphuric acid.*

(**b**) *Erythrosin indicator.*—Dissolve 0.1 gram of the sodium salt in 1 liter of water.

(**c**) *Chloroform.*—Neutral to erythrosin.

81 DETERMINATION.

Measure 100 cc. of the water into a 250 cc. white, glass-stoppered bottle, add 2.5 cc, of the erythrosin and 5 cc. of the chloroform, add N/50 sulphuric acid in small quantities, shaking the bottle vigorously after each addition of the acid. The rose color gradually disappears and is finally discharged by 1 or 2 drops of the acid. A white paper held back of the bottle facilitates the detection of the end point. Multiply the number of cc. of N/50 sulphuric acid used by 10 to obtain the number of parts per million of alkalinity in terms of calcium carbonate.

ALKALINITY.—After Boiling.—Official.

82 REAGENTS.

Described under **80**.

83 DETERMINATION.

Boil 100 cc. of the water in a porcelain dish gently for 30 minutes. Cool, transfer to a 100 cc. volumetric flask and fill to the mark with recently boiled and cooled water. Filter through a dry paper and determine the alkalinity of the filtrate as directed under **81**, making the proper calculation for the aliquot employed and calculating in terms of calcium carbonate the parts per million of alkalinity after boiling.

TOTAL HARDNESS[9].—OFFICIAL.

84 REAGENTS.

(**a**) *Soda reagent.*—Prepare an approximately N/10 solution, using equal parts of sodium hydroxid and sodium carbonate.

(**b**) *N/20 sulphuric acid.*

85 DETERMINATION.

Add sufficient N/20 sulphuric acid to 200 cc. of the sample, contained in a 500 cc. Pyrex or similar glass Erlenmeyer flask, to neutralize the alkalinity, the amount required for this being calculated from the results obtained as directed under **81**. Measure 200 cc. of water into a similar flask. Then treat the contents of each flask in the following manner: Boil 15 minutes to expel free carbon dioxid. Add 25 cc. of the soda reagent. Boil 10 minutes, cool, rinse into 200 cc. graduated flasks and dilute to 200 cc. with boiled water. Filter, rejecting the first 50 cc., and titrate 50 cc. of each filtrate with N/20 sulphuric acid in the presence of methyl orange or erythrosin indicator. The total hardness in parts per million of calcium carbonate is equal to 50 times the difference between the cc. of N/20 sulphuric acid used in titrating the aliquot of the blank and the aliquot of the sample.

86 PERMANENT OR NON-CARBONATE HARDNESS.—OFFICIAL.

The difference between the alkalinity before boiling, **81**, and the total hardness, **85**, is the permanent or non-carbonate hardness expressed as parts per million of calcium carbonate.

IRRIGATING WATER.

87 GENERAL METHODS.—OFFICIAL.

Determine the solids in solution, chlorin, carbonic and bicarbonic acids, sulphuric acid, calcium and magnesium as directed under **7**, **19**, **39**, **48**, **45** and **47**, respectively. To make the hypothetical combination, calculate calcium and magnesium to the acid ions in the following order: bicarbonic, sulphuric and chlorin. Then calculate the remaining acid ions, including carbonic, to the corresponding salts of sodium.

BLACK ALKALI.—OFFICIAL.

88 REAGENTS.

(**a**) *N/50 sodium carbonate.*—One cc. of this solution is equivalent to 0.00106 gram of sodium carbonate.

(**b**) *N/50 sulphuric acid.*—One cc. of this solution is equivalent to 0.0010 gram of calcium carbonate or 0.00136 gram of calcium sulphate.

(**c**) *Erythrosin indicator.*—Dissolve 0.25 gram of the sodium salt in 1 liter of water.

(**d**) *Chlorofrom.*—Neutral to erythrosin.

89 DETERMINATION.

Transfer 200 cc. of the water to a platinum or silver dish, add 50–100 cc. of N/50 sodium carbonate, according to the amount of soluble salts of calcium and magnesium present, and evaporate to dryness. Rub up the residue with carbon dioxid-free water.

For this purpose water should be vigorously boiled until approximately one-third of the original volume is evaporated, then cooled and stoppered. An ordinary laboratory wash bottle should not be used to transfer the residue, as the carbon dioxid from the breath of the operator is sufficient to vitiate the results.

Transfer to a 100 cc. graduated flask, make up to the mark, shake thoroughly, and allow to stand until clear (12–15 hours). Remove 50 cc. of the clear, supernatant liquid, equivalent to one-half of the original quantity of water and sodium carbonate added, and transfer to a glass-stoppered flask or stoppered titration bottle, of 250 cc. capacity, of clear glass, without any tinge of pink. Add 5 cc. of the chloroform and 1 cc. of the erythrosin, and titrate with the standard acid until the color disappears. Shake the solution vigorously after each addition of the acid; the chloroform produces a milky appearance which makes the reading of the end point sharp and certain.

(1) If less sulphuric acid is required than is equivalent to one-half of the sodium carbonate added, due to some of the sodium carbonate reacting with soluble salts of calcium and magnesium, the solution originally contained no black alkali in excess, but rather an excess of the so-called permanent or non-carbonate hardness. It is customary to express the hardness in terms of calcium carbonate or calcium sulphate. With irrigating waters the latter form is to be preferred. Therefore, the difference between the number of cc. of the sulphuric acid required and one-half of the number of cc. of the sodium carbonate added, multiplied by the factor 0.00136, gives the equivalent of calcium sulphate in 100 cc. of the water.

(2) If more sulphuric acid is required than that equivalent to one-half of the sodium carbonate added, black alkali was originally present in the solution, and the difference in cc., multiplied by the factor 0.00106, gives the black alkali in terms of sodium carbonate in 100 cc. of water.

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IV. TANNING MATERIALS.—TENTATIVE.

EXTRACTS.

1 PREPARATION OF SOLUTION.

(a) *Solid extracts.*—Grind solid extracts in a large porcelain mortar, so that the material will pass through a 10 mesh sieve, mix thoroughly and weigh out a quantity containing 3.75–4.25 grams of tannin. This should be done as rapidly as possible to avoid change in moisture content. Pour into 100 cc. of water at 85°C., place on a steam bath and stir until a homogeneous solution is obtained. Transfer to a 1 liter flask with 800 cc. of water at 85°C. Allow to cool overnight at a temperature not below 19°C., bring to 20°C. by placing the flask in water, the temperature of which is not below 19°C., and make up to 1 liter.

(b) *Fluid extracts.*—Allow fluid extracts to come to room temperature and mix thoroughly. Weigh out rapidly a quantity containing 3.75–4.25 grams of tannin. Dissolve by washing into a 1 liter flask with 900 cc. of water at 85°C. Allow to cool and make up to 1 liter at 20°C., as described under **1** **(a)**.

After the preparation of the solutions, proceed at once with the analysis.

2 TOTAL SOLIDS.

Thoroughly mix the prepared solution, pipette at once 100 cc. into a tared flat-bottomed glass dish, 2¾–3 inches in diameter, and (1) evaporate and dry for 16 hours in a combined evaporator and dryer[1] at 98°–100°C.; or, (2) after evaporating on the steam bath, dry for 12 hours on the bottom of a water oven at 98°–100°C. Remove immediately to desiccators containing sulphuric acid (place no more than 2 dishes in 1 desiccator) and weigh rapidly when cooled. Calculate the percentage of total solids.

SOLUBLE SOLIDS.

3 PREPARATION OF FILTER.

The kaolin used should be neutral to phenolphthalein and should not yield more than 1 mg. of soluble solids per 100 cc. of filtrate of a 1 per cent suspension after an hour's digestion at 20°C.

Add about 75 cc. of the solution, as prepared under **1**, to 1 gram of the kaolin in a beaker. Stir and pour immediately into a single, 15 cm. No. 590, S. & S. or No. 1F. Swedish folded filter. Return the filtrate to the paper when approximately 25 cc. have run through, repeat the operation for an hour, thus transferring all the kaolin to the paper. At the end of an hour, discard the solution on the filter by siphoning it off, disturbing the kaolin as little as possible. An ordinary wash bottle serves well for this purpose.

4 DETERMINATION.

Bring about 150 cc. of the original solution, as prepared under **1**, to exactly 20°C. Fill the filter, prepared as under **3**, with this solution and discard the filtrate until it runs through clear. Keep the filter full, the temperature of the filtering solution at 20°–25°C., and the funnel and receiving vessel covered. Pipette at once 100 cc. of the clear filtrate into a tared dish, evaporate and dry as directed under **2**. Calculate the percentage of soluble solids.

5 INSOLUBLE SOLIDS.

The difference between the percentage of the total solids and the percentage of soluble solids is the percentage of the substance insoluble in water at 20°–25°C.

NONTANNINS.

6 REAGENTS.

Hide powder.—This should be of woolly texture, well delimed, and 10 grams of the water-free powder should require 12–13 cc. of N/10 sodium hydroxid to neutralize it.

Calculate the amount of air-dry hide powder which will be required for the number of determinations to be made, on a basis of 13 grams of air-dry hide powder for each determination. Increase this calculated amount by 35 grams of dry hide powder to provide a sufficient amount for all the determinations.

Thoroughly digest the total amount of hide powder with 10 times its weight of water. Then for each gram of the air-dry hide powder, so digested, add 1 cc. of 3 per cent chrome alum solution; and *either* agitate frequently for several hours and let stand overnight *or* agitate in some form of mechanical shaker for an hour. Transfer to a strong linen filter and squeeze thoroughly. Remove from the filter and digest for 15 minutes with a quantity of water equal to 15 times the weight of the dry hide powder employed. Filter and squeeze to approximately 73 per cent of water, using a press if necessary. Very strong pressure is required to reduce the water content below 70 per cent. Repeat the digestion and filtration 3 times. Determine moisture in 20 grams of the squeezed hide powder as directed under **2**.

7 DETERMINATION.

Place 46 grams of the wet hide powder in a suitable container of about 300 cc. capacity, add 200 cc. of the tanning solution, as prepared under **1**, and shake for 10 minutes in a mechanical shaker. Squeeze immediately through linen, add 2 grams of kaolin, as used under **3**, to the filtrate which contains the nontannins, stir and filter through a single, folded 18.5 cm. filter paper (No. 1F. Swedish, preferred), refiltering until the filtrate is clear. The filtrate should give no precipitate with a gelatin-salt solution (1% gelatin and 10% salt). Pipette 100 cc. of the filtrate into a tared dish and evaporate as directed under **2**. Correct the weight of the nontannin residue for the dilution caused by the water retained in the wet hide powder. Calculate the percentage of nontannins.

8 TANNIN.

The difference between the percentage of the soluble solids and the percentage of nontannins is the percentage of tannin.

DETECTION OF SULPHITE-CELLULOSE.

9 REAGENTS.

Sulphite-cellulose solution.—Dissolve 0.5 gram of the total solids, derived from sulphite-cellulose, in 1 liter of water and add sufficient tanning material, free from sulphite-cellulose, to give a concentration of 3.75–4.25 grams of tannin per liter.

10 DETERMINATION.

Place 5 cc. of the tanning solution, prepared as under **1**, in a test tube; add 0.5 cc. of anilin and shake well; then add 2 cc. of concentrated hydrochloric acid and mix again. Compare the precipitate formed with that produced when the above sulphite-cellulose solution is similarly treated. Sulphite-cellulose is held to be present, in the predetermined absence of the synthetic tanning material, Neradol-D, if the volume of the precipitate approximately equals or exceeds that of the comparison solution.

LIQUORS.

11 PREPARATION OF SOLUTION.

Dilute the liquor with water at room temperature to contain approximately 0.7 gram of solids in 100 cc. of solution. If the liquor does not give a proper solution with water at room temperature, it may be diluted with water at 80°C., and then cooled to 20°C. as directed under **1** (**a**).

12 TOTAL SOLIDS.

Proceed as directed under **2**.

13 SOLUBLE SOLIDS.

Proceed as directed under **4**.

14 NONTANNINS.

Proceed as directed under **7**, using the amount of wet chromed hide powder which will give the ratio between the tannin and hide powder shown in the following table:

TANNIN RANGE PER 100 CC.	DRY HIDE POWDER PER 200 CC.
gram	*grams*
0.35—0.45	9.0—11.0
0.25—0.35	6.5— 9.0
0.15—0.25	4.0— 6.5
0.00—0.15	0.0— 4.0

TOTAL ACIDITY.

15 REAGENTS.

(**a**) *Hematin solution.*—Digest 0.5 gram of hematin in 100 cc. of cold neutral 95 per cent alcohol.

(**b**) *Gelatin solution.*—Dissolve 10 grams of gelatin in hot water, cool, add 25 cc. of 95 per cent alcohol and dilute. If the gelatin solution is acid or alkaline, neutralize with N/10 sodium hydroxid or N/10 acetic acid, respectively, using hematin solution as indicator and make up to 1 liter.

(**c**) *Kaolin.*—Digest with dilute hydrochloric acid, wash until it complies with the tests given under **3**, dry and preserve in a tightly stoppered bottle.

(**d**) *N/10 sodium hydroxid.*

16 DETERMINATION.

Add 25 cc. of the gelatin solution to 25 cc. of the tanning liquor in a stoppered cylinder, dilute with water to 250 cc., add 15 grams of the kaolin and shake vigorously. Allow to settle for at least 15 minutes, remove 30 cc. of the supernatant liquid, dilute with 50 cc. of water and titrate with N/10 sodium hydroxid, using the hematin solution as indicator. Each cc. of N/10 sodium hydroxid is equivalent to 0.2 per cent of acid, calculated as acetic, in the liquor.

RAW AND SPENT MATERIALS.

(Under raw materials are included woods, barks, leaves, etc.)

17 MOISTURE IN SAMPLE AS RECEIVED.

Cut or break up large pieces and mix the sample rapidly to avoid change in moisture content. Dry as directed under **2**, a suitable weighed quantity, dependent upon the physical condition and moisture content of the sample.

18 PREPARATION OF SAMPLE.

Dry the remainder of the sample at a temperature not above 60°C., and grind to pass through a 20 mesh sieve.

19 MOISTURE IN PREPARED SAMPLE.

Take 10 grams of the sample prepared in **18**, dry as directed under **2**, and calculate all results to an "as received", "air dry", or "moisture free" basis as desired.

20 EXTRACTION.

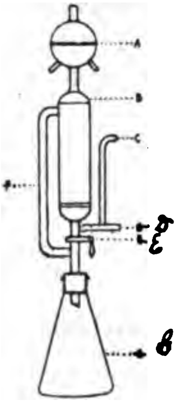

FIG. 2. METAL EXTRACTOR USED FOR EXTRACTING TANNING MATERIALS.

(For spent materials approximate the following quantities as closely as possible.)

Place a quantity of the dried sample, containing 3.75–4.25 grams of tannin, in a beaker and wet thoroughly with hot water. Place a perforated porcelain plate in a tin-lined copper extractor of the general form shown in Fig. 2, and on the plate place a layer of cotton and wet thoroughly with water. Connect the extractor with an 800 cc. Erlenmeyer flask (*G*), open the stop-cock (*E*) and close the outlets (*C*) and (*D*). Pour into the extractor the material to be extracted, washing it into the extractor with hot water. Return the percolate through the extractor until it is practically clear. Place a layer of cotton on top of the material. Close the stop-cock (*E*), connect with an 800 cc. Erlenmeyer flask containing about 650 cc. of water, connect (*D*) by a delivery tube with a liter graduated collecting flask, return the total percolate to the extractor and connect by means of the metal cap (*B*) with a block tin condenser (*A*) in such a way that the condensate will drip upon the layer of cotton. Boil the water in the flask, and collect 400–500 cc. of percolate from the side tube (*D*). Open the stop-cock (*E*) and close the side tube (*D*), add water to the flask (*G*), if necessary, until it contains about 250 cc. and extract for 5 hours. Remove the extract, add 200 cc. of water to the boiling flask and continue the extraction for 9 hours. Throughout the extraction heat at such a rate that approximately 330 cc. of water will be condensed per hour. Combine all the extracts in the graduated liter flask in which the first percolate was received. Heat to 80°C., cool as directed under **1** (**a**) and make up to the mark.

21 ANALYSIS OF THE EXTRACT.

Proceed as directed under **2-8**, inclusive. If more dilute solutions than the directions specify are employed in the determination of nontannins, the amount of hide powder used is reduced, as directed under **14**.

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V. LEATHERS.—TENTATIVE.

VEGETABLE TANNED LEATHER.

1 PREPARATION OF SAMPLE.

Grind the sample, without undue heating, and pass through a 10 mesh sieve. The ground sample must not contain hard lumps. Plane heavily greased leathers (containing more than 20 per cent fat) into very thin shavings. Spread out the prepared sample and allow it to return to atmospheric moisture condition; mix thoroughly, and place in tightly covered containers.

2 MOISTURE.

Place 10 grams of the sample, as prepared under **1**, in a tared, wide, shallow, weighing bottle (or a similar dish which can be covered tightly), and dry in a water oven for 15 hours at 98°-100°C. Cover the weighing bottle, cool in a desiccator containing sulphuric acid and weigh. The moisture present in the leather as received may be determined by cutting it quickly into small pieces and drying without grinding as directed above.

3 TOTAL ASH.

Incinerate slowly 5 grams of the sample, as prepared under **1**, at a dull red heat. If difficulty is experienced in burning off the carbon, leach the residue with hot water, filter on an ashless filter, dry and ignite the filter and residue, add the filtrate, evaporate to dryness and ignite. Cool in a desiccator containing sulphuric acid and weigh.

The ash may be examined for acids and bases by any suitable method. Aluminium, magnesium, sodium, barium, calcium and lead are the bases, and hydrochloric and sulphuric acids are the acids which it may be necessary to determine.

4 INSOLUBLE ASH.

Incinerate slowly the residue from the extraction of water-soluble material, obtained in **6** or **7**, until all the carbon is burned off, cool in a desiccator containing sulphuric acid and weigh.

5 FATS.

Place, without packing, 15 grams of the leather, as prepared under **1**, in a Soxhlet or Johnson extractor with a layer of fat-free cotton above and below the sample. Extract 8-10 hours with petroleum ether distilling between 50° and 80°C. Heavily greased leathers (containing 15 per cent or more fat) will require the maximum time. Remove the receiving flask, evaporate the petroleum ether on the steam bath and dry the fat residue for 3 hours in a water oven at 98°-100°C., cool in a desiccator and weigh. Repeat the drying in the water oven for periods of 1-1½ hours, cooling and weighing as before, until no further loss in weight occurs. Retain the leather residue from the fat extraction for the extraction of water-soluble material as directed under **6**.

EXTRACTION OF WATER-SOLUBLE MATERIAL.

6 *Method I.*

Evaporate the petroleum ether from the fat-free leather, obtained under **5**, and moisten thoroughly with 100-150 cc. of water. Place a layer of cotton in the bottom of a Soxhlet extractor designed for making extractions at temperatures below 100°C.

The water jacket of an extractor of this kind surrounds that portion of the apparatus containing the sample but does not enclose the side tube which carries the hot vapors to the condenser.

Transfer the moistened fat-free leather to the extractor and cover it with another layer of cotton to avoid siphoning off solid particles. Maintain the temperature of the jacket surrounding the Soxhlet at 50°C. (1) Pour 200 cc. of water (including that used in moistening the leather) into the Soxhlet and allow it to siphon into the flask below, then heat and extract for an hour. Remove the flame and transfer the extract to a liter graduated flask. Then add water and continue the extraction as directed below, removing and transferring the extract to the liter flask before each fresh addition of water.

(2) Add 175 cc. of water and extract for 2 hours.
(3) Add 175 cc. of water and extract for 3 hours.
(4) Add 175 cc. of water and extract for 4 hours.
(5) Add 175 cc. of water and extract for 4 hours.

Transfer the last portion of the extract to the graduated flask. This gives 14 hours' extraction and an extract which does not exceed 1 liter in volume. Dilute to 1 liter at room temperature and mix thoroughly.

7 *Method II.*

(This method is the same in principle as the official method of the American Leather Chemists Association[1].)

Extract 30 grams of the leather prepared as directed under **1** with petroleum ether as directed under **5**; evaporate the solvent from the leather and digest overnight with about 200 cc. of water. Transfer the leather and extract to a percolator. Continue the extraction by percolating with water at 50°C. Collect 2 liters of percolate, regulating the flow of water at such a rate that 2 liters will be collected in 3 hours. Dilute to volume at room temperature and mix thoroughly.

To the extract, prepared according to **6** or **7**, add a few drops of toluol to prevent fermentation of sugars, and reserve for the determination of glucose, total solids, soluble solids and nontannins.

GLUCOSE.

8 PREPARATION OF SOLUTION.

To 200 cc. of the leather extract, as prepared under **6** or **7**, add 25 cc. of a saturated solution of neutral lead acetate, mix thoroughly and filter at once through a dry, folded filter, returning the first portions of the filtrate to the filter until the filtrate becomes clear. Keep the containers and the funnel covered during these operations. Without waiting for the entire filtrate to run through add 10–12 grams of solid potassium oxalate, shake frequently during 15–20 minutes and filter through a dry, folded filter returning the first runnings to the filter until the filtrate runs clear. Pipette 150 cc. of the last filtrate into a 600 cc. Erlenmeyer flask, add 5 cc. of concentrated hydrochloric acid and boil under a reflux condenser for 2 hours. Cool, neutralize with solid sodium carbonate, using a few drops of methyl orange as indicator, transfer to a 200 cc. volumetric flask and complete to volume with water. Filter through a double filter and return the first runnings until the filtrate becomes perfectly clear. Determine the dextrose in the filtrate immediately.

9 DETERMINATION.

Determine dextrose in 50 cc. of the solution, as prepared under **8**, equivalent to 0.5 gram of leather, according to **VII, 25**, weighing directly as cuprous oxid, **VII, 26**, and express the result as glucose.

10 TOTAL SOLIDS.

Determine as directed under **IV, 2**.

11 SOLUBLE SOLIDS.

Determine as directed under **IV, 4**.

12 NONTANNINS.

Determine as directed under **IV, 7**.

13 SOLUBLE TANNIN.

The difference between the percentage of the soluble solids and the corrected nontannins is the percentage of tannin.

14 NITROGEN.

Determine as directed under **I, 21**.

15 HIDE SUBSTANCE.

Multiply the percentage of nitrogen by 5.62. The result will be the percentage of hide substance present.

16 COMBINED TANNIN.

Deduct the sum of the percentages of moisture, under **2**, insoluble ash, under **4**, fats, under **5**, soluble solids, under **11**, and hide substance, under **15**, from 100. The result will be the percentage of combined tannin.

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VI. INSECTICIDES AND FUNGICIDES.

GENERAL METHOD.

1 PREPARATION OF SAMPLE.—TENTATIVE.

Thoroughly mix all samples before analysis. Make water-soluble arsenic determinations on samples as received without further pulverization or drying. In the case of lye, sodium cyanid or potassium cyanid, weigh large quantities in weighing bottles and analyze aliquots of the aqueous solutions.

PARIS GREEN.

2 MOISTURE.—TENTATIVE.

Dry 2 grams at 105°–110°C. for 5 hours and express the loss in weight as moisture.

TOTAL ARSENIC[1].—OFFICIAL.

(Arsenic, present as arsenate, is titrated as arsenious oxid.)

3 REAGENTS.

(a) *Starch indicator.*—Mix about 0.5 gram of finely powdered potato starch with cold water to a thin paste; pour into about 100 cc. of boiling water, stirring constantly, and discontinue heating immediately after the paste is added.

(b) *Standard arsenious oxid solution.*—Dissolve 2 grams of pure arsenious oxid in a beaker by boiling with about 150–200 cc. of water containing 10 cc. of concentrated sulphuric acid, cool, transfer to a 500 cc. graduated flask and dilute to the mark.

(c) *Standard iodin solution.*—Prepare an approximately N/20 solution as follows: Mix intimately 6.35 grams of pure iodin with twice its weight of pure potassium iodid. Dissolve in a small amount of water, filter and dilute the filtrate to 1 liter in a liter graduated flask. Standardize against (b) as follows: Pipette 50 cc. of the arsenious oxid into an Erlenmeyer flask, dilute to the same volume as that of the aliquot used for the titration in the actual determination, neutralize with sodium bicarbonate, add 4–5 grams in excess and add the standard iodin solution from a burette, shaking the flask continuously, until the yellow color disappears slowly from the solution, then add 5 cc. of the starch indicator and continue adding the iodin solution, drop by drop, until a permanent blue color is obtained. Calculate the value of the standard iodin solution in terms of *arsenious* oxid (As_2O_3) and *arsenic* oxid (As_2O_5). For the conversion of *arsenious* oxid (As_2O_3) to *arsenic* oxid (As_2O_5) multiply by 1.1617. Occasionally restandardize the iodin against the standard arsenious oxid solution.

4 APPARATUS.

The apparatus used is shown in Fig. 3. The distillation flask rests on a metal gauze which fits over a circular hole in a heavy sheet of asbestos board, which in turn extends out far enough to protect the sides of the flask from the direct flame of the burner. The first flask which receives the distillate is of 500 cc. capacity and contains not more than 40 cc. of water, the second is of 1000 cc. capacity and contains 100 cc of water. The volume of water in the first flask should not exceed 40 cc., otherwise a compound of arsenic will separate when the hot acid vapors strike the cold water, which can not readily be got into solution without danger of loss of arsenious chlorid.

Both these flasks should be placed in a pan and kept surrounded with water and cracked ice. The third flask, containing sufficient water to seal the end of the glass tube leading into it, is added as a precaution. It is almost never found to contain arsenic.

5 DETERMINATION.

Weigh an amount of the sample equal to the arsenious oxid equivalent of 250 cc. of the standard iodin solution, and wash into the distillation flask by means of 100 cc. of concentrated hydrochloric acid (sp. gr. 1.19). Add 5 grams of cuprous chlorid (Cu_2Cl_2) and distil.

When the volume in the distillation flask is reduced to about 40 cc., add 50 cc. of concentrated hydrochloric acid by means of the dropping funnel and continue the distillation until 200 cc. of the acid distillate have passed over. Then wash down the condenser and all the connecting tubes carefully, transfer these washings and the contents of the 3 Erlenmeyer flasks to a liter graduated flask and dilute to the mark. Mix thoroughly, pipette 400 cc. into an Erlenmeyer flask and nearly neutralize with a saturated solution of sodium or potassium hydroxid, using a few drops of phenolphthalein as indicator, keeping the solution well cooled. If the neutral point is passed, add hydrochloric acid until again slightly acid.

Continue as directed under **3** (**c**) beginning with "neutralize with sodium bicarbonate". The number of cc. of iodin used in this titration represents directly the total per cent of arsenic in the sample expressed as arsenious oxid (As_2O_3).

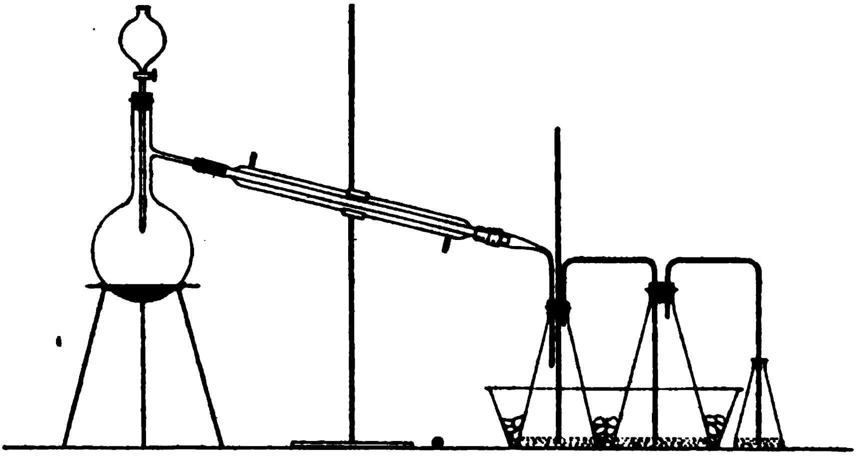

FIG. 3. APPARATUS FOR DISTILLATION OF ARSENIOUS CHLORID.

TOTAL ARSENIOUS OXID.

(The following methods determine only the arsenic present in the form of the *-ous* oxid, As_2O_3. They also determine any antimony which may be present in the form of Sb_2O_3. Ferrous and cuprous salts vitiate the results.)

Method I[1].—*Tentative.*

6 REAGENTS.

The reagents and solutions used are described under **3**.

7

DETERMINATION.

Weigh an amount of the sample equal to the arsenious oxid equivalent of 100 cc of the standard iodin solution, wash into an Erlenmeyer flask with 10–15 cc. of dilute hydrochloric acid (1 to 1), followed by about 100 cc. of water and heat on the steam bath only as long as is necessary to complete solution, at a temperature not exceeding 60°C. Cool, neutralize with sodium bicarbonate, add 4–5 grams in excess and then sufficient 25 per cent ammonium chlorid solution to dissolve the precipitated copper. Dilute somewhat and titrate as directed under **3** (**c**). A correction must be applied for the amount of iodin solution necessary to produce a blue color with starch in the presence of copper (using an equivalent weight of copper sulphate). The corrected number of cc. of the standard iodin solution used represents directly the per cent of arsenious oxid (As_2O_3) in the sample.

8

Method II[3].—*Tentative.*

Proceed as directed under **7**, using dilute sulphuric acid (1 to 4) instead of dilute hydrochloric. The solution in this case may be heated to boiling.

SODIUM ACETATE-SOLUBLE ARSENIOUS OXID.[4]—TENTATIVE.

9

REAGENTS.

(**a**) *Sodium acetate solution.*—Prepare a solution containing 12.5 grams of the crystallized salt ($CH_3COONa3H_2O$) in each 25 cc.

The other reagents are described under **3**.

10

DETERMINATION.

Place 1 gram of the sample in a 100 cc. flask and boil for 5 minutes with 25 cc of the sodium acetate. Dilute to the mark, shake and pass through a dry filter paper. Titrate an aliquot of this filtrate as directed under **3** (**c**). Calculate the amount of arsenious oxid (As_2O_3) present and express the result as per cent of sodium acetate-soluble arsenious oxid.

WATER-SOLUBLE ARSENIOUS OXID.—TENTATIVE.

11

REAGENTS.

The reagents and solutions used are described under **3**.

12

DETERMINATION.

To 1 gram of the sample in a liter Florence flask add 1 liter of recently boiled water which has been cooled to exactly 32°C. Stopper the flask and place in a water bath kept at 32°C. by means of a thermostat. Digest for 24 hours, shaking hourly for 8 hours during this period. Filter through a dry filter and titrate 250 cc. of the filtrate as directed under **3** (**c**). Correct for the amount of the standard iodin necessary to produce the same color, using the same reagents and volume. Calculate the amount of arsenious oxid (As_2O_3) present and express the results as per cent of water-soluble arsenious oxid

TOTAL COPPER OXID.

13

Electrolytic Method.—Official.

Treat 2 grams of the sample in a beaker with 100 cc. of water and about 2 grams of sodium hydroxid and boil thoroughly until all the copper is precipitated as cuprous oxid. Filter, wash well with hot water, dissolve the precipitate in hot dilute nitric acid, cool, transfer to a 250 cc. graduated flask and dilute to the mark. (1) Use 50–100 cc. of this solution for the electrolytic determination of copper as directed under **VII, 32**, and calculate to per cent of cupric oxid; or, (2) Electrolyze the aliquot in a weighed 150 cc. platinum dish, using a rotating spiral anode and a current of about 3 amperes. After all the copper is deposited (requiring about 30 minutes), wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven, weigh and calculate to per cent of cupric oxid.

14 *Thiosulphate Methods*[5].—*Official.*

Determine copper in another aliquot of the nitric acid solution of copper oxid, under **13**, by titrating with N/20 thiosulphate solution, as directed under **VII**, **28**, and calculate to per cent of cupric oxid.

LONDON PURPLE.

15 MOISTURE.—TENTATIVE.

Proceed as directed under **2**.

TOTAL ARSENIOUS OXID[6].—OFFICIAL.

16 REAGENTS.

The reagents and solutions used are described under **3**.

17 DETERMINATION.

Dissolve 2 grams of the sample in a mixture of about 80 cc. of water and 20 cc. of concentrated hydrochloric acid at a temperature of 60°–70°C.; filter and wash until the combined filtrate and washings measure 250 cc. Treat 100 cc. of this solution with sodium bicarbonate in excess, transfer to a 500 cc. volumetric flask and make up to the mark, adding a few drops of ether to destroy the bubbles. Mix thoroughly and pass through a dry filter. Titrate 250 cc. of the filtrate as directed under **3** (**c**) and calculate the per cent of arsenious oxid.

TOTAL ARSENIC OXID[7].—OFFICIAL.

18 REAGENTS.

The reagents and solutions used are described under **3**.

19 DETERMINATION.

Boil, on a hot plate or over a low flame, 2 grams of the sample with 5 cc. of concentrated nitric acid and 20 cc. of concentrated sulphuric acid in a Kjeldahl digestion flask or a covered casserole. After 10–15 minutes add fuming nitric acid or powdered sodium nitrate, in small quantities at a time, until all organic matter is destroyed and the solution is practically colorless. Cool, add about 50 cc. of water (to decompose any nitro-sulphuric acid formed) and heat again until all nitric acid fumes are expelled. Cool, transfer to a 250 cc. volumetric flask, make up to the mark with water, mix thoroughly and filter through a dry filter.

Transfer 50 cc. of this filtrate to a 400 cc. Erlenmeyer flask, dilute with water to 100 cc., add 1 gram of potassium iodid[8], heat to boiling and evaporate to about 40 cc. (not less). Cool, dilute to 150–200 cc., and remove the excess of iodin with N/20 sodium thiosulphate. In case the solution is slightly colored from organic matter or from any cause other than free iodin, add the thiosulphate until it is nearly colorless, then a few drops of the starch indicator and continue adding the thiosulphate slowly until the blue color just disappears. Continue at once as directed under **3** (**c**) beginning with "neutralize with sodium bicarbonate". Subtract from this reading the number of cc. of the standard iodin solution corresponding to the arsenious oxid obtained in **17**. Calculate the per cent of arsenic oxid in the sample.

20 WATER-SOLUBLE ARSENIOUS OXID.—TENTATIVE.

Proceed as directed under **12**, slightly acidifying the aliquot employed with hydrochloric acid before adding the excess of sodium bicarbonate.

WATER-SOLUBLE ARSENIOUS OXID.—TENTATIVE.

21 REAGENTS.

The reagents and solutions used are described under **3**.

22 DETERMINATION.

Transfer an aliquot, 250 cc., of the water extract, from **20**, to a casserole, add 5 cc. of concentrated sulphuric acid, evaporate to a small volume and heat on a hot plate till white fumes of sulphuric acid appear. Cover the casserole and add 1–2 cc. of fuming nitric acid and again heat till the appearance of white fumes. Cool, add a little water and, in order to expel the last traces of nitric acid, once more evaporate till white fumes appear. Cool, dilute to about 100 cc. with water, add 1 gram of potassium iodid[8] and sufficient sulphuric acid to make the total amount present about 5 cc. Boil until the volume is reduced to about 40 cc. Cool, dilute to about 200 cc., remove the excess iodin with N/20 sodium thiosulphate and proceed as directed under **3** (**c**) beginning with "neutralize with sodium bicarbonate". Correct for the amount of the standard iodin solution necessary to produce the same color, using the same reagents and volume. Subtract from the corrected titration reading the number of cc. of the standard iodin solution corresponding to the arsenious oxid, obtained under **20**. Calculate the per cent of arsenic oxid present.

LEAD ARSENATE.

23 MOISTURE.—TENTATIVE.

(**a**) *Powder*.—Dry 2 grams to constant weight at 105°–110° C. and report the loss in weight as moisture.

(**b**) *Paste*.—Proceed as under (**a**), using 50 grams. Grind the dry sample to a fine powder, mix well, transfer a small portion to a sample bottle and again dry for 1–2 hours at 105°–110°C., and use this anhydrous material for the determination of total lead oxid and total arsenic.

TOTAL LEAD OXID.

24 *Method I*[9].—*Official.*

Heat, on a hot plate, 0.6906 gram of the dry powdered sample with about 25 cc. of dilute nitric acid (1 to 4) in a 600 cc. beaker. If necessary, remove any insoluble residue by filtration. Dilute to at least 400 cc., heat nearly to boiling, add ammonium hydroxid to incipient precipitation, then dilute nitric acid (1 to 10) to redissolve the precipitate, adding 1–2 cc. in excess. Pipette into this solution, kept almost boiling, 50 cc. of a hot 10 per cent potassium chromate solution, stirring constantly. Decant while hot through a weighed Gooch, previously heated at 140°–150°C., wash several times by decantation and then on the filter with boiling water until the washings are colorless. Dry the lead chromate at 140°–150°C. to constant weight. The weight of lead chromate. multiplied by 100, gives the per cent of lead monoxid (PbO) in the dried sample. The lead chromate precipitate may contain a small amount of lead arsenate which causes slightly high results. This error rarely amounts to more than 0.1–0.2 per cent.

Method II[10].—*Tentative.*

(Not applicable in the presence of calcium.)

25 REAGENTS.

Acidified alcohol.—Mix water 100 parts; 95 per cent alcohol 200 parts; and concentrated sulphuric acid 3 parts by volume.

26 DETERMINATION.

Heat, on a hot plate, 0.7360 gram of the dry powdered sample with about 25 cc. of dilute nitric acid (1 to 4) in a porcelain evaporating dish or casserole. Remove any insoluble residue by filtration. Add 3 cc. of concentrated sulphuric acid and evaporate on the hot plate to the appearance of white fumes. It is important that all nitric acid be expelled. Cool, add 50 cc. of water and about 100 cc. of 95 per cent alcohol, let stand several hours (preferably over night) and filter through a weighed Gooch crucible, previously washed with water, the acidified alcohol and 95 per cent alcohol, and dried at 200°C. Wash the precipitate of lead sulphate in the crucible about 10 times with the acidified alcohol and then with 95 per cent alcohol until free from sulphuric acid. Dry at 200°C. to constant weight, keeping the crucible covered to prevent loss by spattering. The weight of the lead sulphate, multiplied by 100, gives the per cent of lead monoxid (PbO) in the dried sample.

TOTAL ARSENIC.

27 *Method I*[1].—*Official.*

Proceed as directed under **5**, using an amount of the sample equal to the arsenic oxid equivalent of 500 cc. of the standard iodin solution and titrating a 200 cc. aliquot of the distillate. The number of cc. of the standard iodin solution used represents directly the total per cent of arsenic in the sample expressed as arsenic oxid (As_2O_5).

Method II[11].—*Official.*

(Not applicable in the presence of antimony.)

28 REAGENTS.

The reagents and solutions used are described under **3**.

29 DETERMINATION.

Dissolve an amount of the powdered sample equal to the arsenic oxid equivalent of 400 cc. of the standard iodin solution, in dilute nitric acid in a porcelain casserole or evaporating dish. Add 5 cc. of concentrated sulphuric acid and heat on the hot plate to copious evolution of white fumes. Cool, add a little water and again evaporate until the appearance of white fumes to assure removal of the last trace of nitric acid. Wash into a 200 cc. graduated flask with water, cool, make up to the mark and filter through a dry filter. Transfer 100 cc. of the filtrate to an Erlenmeyer flask and proceed as directed under **22** beginning with "add 1 gram potassium iodid" to "Subtract from the corrected titration reading". The number of cc. of the standard iodin solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenic oxid (As_2O_5).

WATER-SOLUBLE ARSENIC OXID.—TENTATIVE.

30

REAGENTS.

The reagents and solutions used are described under 3.

31

DETERMINATION.

To 2 grams of the original sample, if a powder, or 4 grams, if a paste, in a liter Florence flask, add 1 liter of recently boiled water which has been cooled to exactly 32°C. Stopper the flask and place in a water bath kept at 32°C. by means of a thermostat. Digest for 24 hours, shaking hourly for 8 hours during this period. Filter through a dry filter, transfer 250–500 cc. of the *clear* filtrate to an Erlenmeyer flask, add 3 cc. of concentrated sulphuric acid and evaporate on a hot plate. When the volume reaches about 100 cc. add 1 gram of potassium iodid, and continue the boiling until the volume is about 40 cc. Cool, dilute to about 200 cc., remove the excess iodin with N/20 sodium thiosulphate, avoiding the use of starch solutions at this point, and proceed as directed under 3 (c) beginning with "neutralize with sodium bicarbonate". Make correction for the amount of iodin solution necessary to produce the same color using the same reagents and volume. Calculate and report as per cent of water-soluble *arsenic* oxid (As_2O_5).

TOTAL ARSENIOUS OXID—TENTATIVE.

32

REAGENTS.

The reagents and solutions used are described under 3.

33

DETERMINATION.

Weigh an amount of the powdered sample equal to the arsenious oxid equivalent of 1 liter of the standard iodin solution, transfer to a 200 cc. graduated flask, add 100 cc of dilute sulphuric acid (15 cc. of concentrated sulphuric acid and 85 cc. of water) and boil for 30 minutes. Cool, make to volume, shake thoroughly and filter through a dry filter. Nearly neutralize 100 cc. of the filtrate with a strong solution of sodium hydroxid, using a few drops of phenolphthalein as indicator. If the neutral point is passed, make acid again with dilute sulphuric acid. Continue as directed under 3 (c) beginning with "neutralize with sodium bicarbonate". The number of cc. of iodin used in this titration. multiplied by 0.2, represents directly the per cent of arsenious oxid (As_2O_3) in the sample.

TOTAL ARSENIC OXID.—TENTATIVE.

34

REAGENTS.

(a) *Starch solution.*—Prepare as directed under 3 (a).

(b) *Standard iodin solution.*—Prepare as directed under 3 (c) but calculate in terms of arsenic oxid (As_2O_5).

(c) *Standard thiosulphate solution.*—Prepare an approximately N/20 solution as follows: Dissolve 13 grams of crystallized sodium thiosulphate in recently boiled and cooled water, filter and make up to 1 liter with recently boiled and cooled water. Standardize as follows: Dissolve about 0.7 gram of lead hydrogen arsenate ($PbHAsO_4$) in 50 cc. of concentrated hydrochloric acid in an Erlenmeyer flask.

Pure $PbHAsO_4$ may be prepared by pouring a solution of lead nitrate into a solution of potassium dihydrogen arsenate (KH_2AsO_4), which should be in excess. Collect the precipitate by filtration, dissolve it in the smallest possible quantity of boiling dilute nitric acid (1 to 4) and pour the solution into a large quantity of water. Collect the precipitate by filtration and dry at 110°C.

If necessary to effect solution, heat on a steam bath, keeping the flask covered with a watch glass to prevent evaporation of the acid. Cool to 20°–25°C., add 10 cc. of potassium iodid solution (20 grams of potassium iodid dissolved in water and diluted to 100 cc.) and 50 cc. (or more if necessary to produce a clear solution) of ammonium chlorid solution (25 grams of ammonium chlorid dissolved in water and diluted to 100 cc.) and immediately titrate the liberated iodin with the standard thiosulphate. When the color becomes a faint yellow, dilute with about 150 cc. of water and continue the titration carefully, drop by drop, until colorless, using starch solution as an indicator near the end point. From the weight of lead hydrogen arsenate and the number of cc. of sodium thiosulphate solution used, calculate the value of the latter in terms of arsenic oxid (As_2O_5). (As_2O_5 in $PbHAsO_4 = 33.11\%$.)

35 DETERMINATION.

Weigh an amount of the powdered sample equal to the arsenic oxid equivalent of 200 cc. of the standard thiosulphate solution, transfer to an Erlenmeyer flask, add 25–30 cc. of concentrated hydrochloric acid and evaporate to dryness on a steam bath. Then add 50 cc. of concentrated hydrochloric acid and proceed as directed under **34** (c), beginning with "If necessary to effect solution, heat on a steam bath". The number of cc. of thiosulphate solution used in this titration, divided by 2, represents directly the per cent of arsenic oxid (As_2O_5) in the sample.

CALCIUM ARSENATE.

36 TOTAL ARSENIC[1].—OFFICIAL.

Proceed as directed under **5**, using an amount of the powdered sample equal to the arsenic oxid equivalent of 250 cc. of the standard iodin solution. The number of cc. of the standard iodin solution used represents directly the total per cent of arsenic in the sample expressed as arsenic oxid (As_2O_5).

ZINC ARSENITE.

37 TOTAL ARSENIC[1].—OFFICIAL.

Proceed as directed under **5**, using an amount of the powdered sample equal to the arsenious oxid equivalent of 500 cc. of the standard iodin solution and titrating a 200 cc. aliquot of the distillate. The number of cc. of the standard iodin solution used represents directly the per cent of total arsenic in the sample expressed as arsenious oxid (As_2O_3).

38 TOTAL ARSENIOUS OXID.—TENTATIVE.

Proceed as directed under **7** or **8**.

COPPER CARBONATE.

39 COPPER OXID.—OFFICIAL.

Dissolve a weighed quantity of the substance in dilute nitric acid and determine copper as directed under **13** or **14**.

BORDEAUX MIXTURE.

40 MOISTURE.—OFFICIAL.

(a) *Powder.*—Dry 2 grams to constant weight at 105°–110°C. and express the loss in weight as moisture.

(**b**) *Paste.*—Heat about 100 grams in an oven at 90°–100°C. until dry enough to powder readily, and note the loss in weight. Powder this partially dried sample, and determine the remaining moisture in 2 grams as under (**a**). Determine carbon dioxid, as directed under **42**, both in the original paste and in this partially dried sample. Calculate the total moisture by the following formula:

$M = a + (100-a)(b + c) - d$ in which

M = per cent of total moisture in original paste;

a = per cent loss in weight of original paste during first drying;

b = per cent loss in weight of partially dried paste during second drying;

c = per cent of carbon dioxid remaining in partially dried paste after first drying;

d = per cent of total carbon dioxid in original paste.

CARBON DIOXID[12].—OFFICIAL.

41

APPARATUS.

This consists of a 200 cc. Erlenmeyer flask closed with a 2-holed stopper; one hole is fitted with a dropping funnel the stem of which extends almost to the bottom of the flask; the outlet of a condenser, which is inclined upward at an angle of 30° from the horizontal, passes downward through the other hole. The upper end of the condenser is connected with a calcium chlorid tube which in turn is connected with a double U-tube filled in the middle with pumice fragments, previously saturated with copper sulphate solution and subsequently dehydrated, and with calcium chlorid at either end. Then follow two weighed U-tubes for absorbing the carbon dioxid, the first filled with porous soda-lime, and the second, one-third with soda-lime and two-thirds with calcium chlorid, the latter reagent being placed at the exit end of the train. A Geissler bulb, partly filled with sulphuric acid, is attached to the last U-tube to show the rate of gas flow. An aspirator is connected with the Geissler bulb to draw air through the apparatus. An absorption tower filled with soda-lime is connected with the mouth of the dropping funnel to remove carbon dioxid from the air entering the apparatus.

42

DETERMINATION.

Weigh 2 grams of the powder or 10 grams of the paste into the Erlenmeyer flask, add about 20 cc. of water, attach the flask to the apparatus, omitting the two weighed U-tubes, and draw carbon dioxid-free air through the apparatus until the original air is displaced. Then attach the weighed U-tubes in position, as described in **41**, close the stop-cock of the dropping funnel, fill half full with dilute hydrochloric acid (1 to 1), reconnect with the soda-lime tower, and allow the acid to flow into the Erlenmeyer flask, slowly if there is much carbon dioxid, rapidly if there is little. When effervescence diminishes, place a low Bunsen flame under the flask and start a flow of water through the condenser, a slow current of air being allowed to flow through the apparatus at the same time. Maintain a steady but quiet ebullition, and a slow air current through the apparatus. Boil for a few minutes after the water has begun to condense in the condenser, then remove the flame and continue the aspiration of air at the rate of about 2 bubbles per second until the apparatus is cool. Disconnect the tared absorption tubes, cool in the balance case and weigh. The increase in weight is carbon dioxid.

COPPER.

43

Electrolytic Method.–Official.

Dissolve 2 grams of the dry powdered sample in 20 cc. of water and 5 cc. of concentrated nitric acid, dilute to 100 cc., wash into a weighed 150 cc. platinum dish, and electrolyze, using a rotating spiral anode and a current of about 3 amperes. After all the copper is deposited (requiring about 30 minutes), wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven and weigh. Calculate the per cent of copper in the sample.

44 *Thiosulphate Method.—Official.*

Dissolve 2 grams of the dry powdered sample in about 50 cc. of 10 per cent nitric acid, add ammonium hydroxid solution in excess and heat; then, without removing the precipitate which is formed, boil off the excess of ammonia, add 5–10 cc. of acetic acid, cool, add 10 cc. of 30 per cent potassium iodid solution and titrate as directed under **VII, 28**.

BORDEAUX MIXTURE WITH PARIS GREEN.

45 MOISTURE.—OFFICIAL.

Proceed as directed under **40**.

46 CARBON DIOXID.—OFFICIAL.

Proceed as directed under **42**.

COPPER.

47 *Method I.—Tentative.*

Dissolve 2 grams of the dry powdered sample in a few cc. of strong nitric acid, add 25 cc. of a 3 per cent solution of hydrogen peroxid and warm for 5–10 minutes. Make slightly alkaline with ammonium hydroxid and then slightly acid again with dilute nitric acid. Transfer to a weighed 150 cc. platinum dish, add 15–20 cc. of hydrogen peroxid, dilute to 100 cc. and electrolyze, using a rotating spiral anode and a current not exceeding 2 amperes. After the electrolysis has proceeded for about 20 minutes, add to the electrolyte 0.5 gram of ferric sulphate dissolved in a few cc. of water together with a drop or two of nitric acid. After all the copper is deposited, wash the deposit with water by siphoning, then rinse with alcohol, dry for a few minutes in an oven, weigh and calculate the per cent of copper. (Do not pass the current for more than 5–10 minutes after all the copper has been deposited without adding more ferric sulphate solution.)

48 *Method II.—Tentative.*

Treat 1 gram of the dry powdered sample with 20 cc. of water and 5–6 cc. of concentrated nitric acid, heat to boiling, cool, and add a slight excess of concentrated ammonium hydroxid. Wash the solution and precipitate into a weighed platinum dish of about 150 cc. capacity, and electrolyze, using a rotating anode and a current of about 4 amperes and 3–4 volts for about 90 minutes (or until all the copper is deposited). Wash the deposit by siphoning until it is clean, being careful not to use too much wash water. Dissolve the copper in 5 cc. of concentrated nitric acid, dilute to 100 cc. and electrolyze as before, except that all the copper will be deposited in 30 minutes. Wash the deposit with water by siphoning, then rinse with alcohol, dry for a minute or so in an oven, weigh and calculate the per cent of copper.

49 TOTAL ARSENIC[1].—OFFICIAL.

Proceed as directed under **5**, using an amount of the dry powdered sample equal to the arsenious oxid equivalent of 500 cc. of the standard iodin solution. The number of cc. of the standard iodin solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenious oxid (As_2O_3).

TOTAL ARSENIOUS OXID.

50 *Method I.—Tentative.*

Proceed as directed under **7**, using an amount of the dry powdered sample equal to the arsenious oxid equivalent of 200 cc. of the standard iodin solution. Before titrating, all the copper must be in solution. The corrected number of cc. of the standard iodin solution used, divided by 2, represents directly the per cent of total arsenious oxid (As_2O_3) in the sample.

51 *Method II.—Tentative.*

Proceed as directed under **8**.

52 WATER-SOLUBLE ARSENIOUS OXID.—TENTATIVE.

Proceed as directed under **20**, using 2 grams of the sample.

BORDEAUX MIXTURE WITH LEAD ARSENATE.

53 MOISTURE.—OFFICIAL.

Proceed as directed under **40**.

54 CARBON DIOXID.—OFFICIAL.

Proceed as directed under **42**.

55 COPPER.—TENTATIVE.

Proceed as directed under **48**.

56 LEAD OXID[3].—TENTATIVE.

Dissolve the lead peroxid (which will contain a little arsenic) from the anodes used in the copper electrolysis, under **55**, by means of dilute nitric acid and a little hydrogen peroxid, and add to this solution the washings from both electrolyses of copper. Add ammonium chlorid to dissolve any lead sulphate which may have precipitated out and make the solution up to 1 liter. Concentrate a 500 cc. aliquot of this solution to about 300 cc. (all hydrogen peroxid must be expelled from the solution), transfer to a 400 cc. beaker and precipitate the lead as lead chromate as directed under **24**.

57 TOTAL ARSENIC[1].—OFFICIAL.

Proceed as directed under **5**, using an amount of the dry powdered sample equal to the arsenic oxid equivalent of 500 cc. of the standard iodin solution. The number of cc. of the standard iodin solution used, divided by 2, represents directly the per cent of total arsenic in the sample expressed as arsenic oxid (As_2O_5).

58 WATER-SOLUBLE ARSENIC OXID.—TENTATIVE.

Proceed as directed under **31**.

SODIUM AND POTASSIUM CYANIDS.

59 CYANOGEN[13].—OFFICIAL.

Weigh about 10 grams of the sample in a weighing bottle, dissolve in water, and make up to volume in a liter graduated flask. To a 50 cc. aliquot add N/20 silver nitrate, drop by drop, stirring constantly, until 1 drop produces a permanent turbidity. In calculating the results, 1 equivalent of silver is equal to 2 equivalents of cyanogen, according to the following equation:

$$2NaCN + AgNO_3 = NaCNAgCN + NaNO_3.$$

Reserve the titrated solution for the determination of chlorin under **60**.

60 CHLORIN[14].—OFFICIAL.

After completion of the titration for cyanogen, as directed under **59**, add a few cc. of 10 per cent potassium chromate solution as indicator and titrate with N/20 silver nitrate until the red-brown color of silver chromate appears.

The first titration with silver nitrate represents the cyanogen present according to the equation under **59**. The second titration represents the cyanogen and chlorin according to the following equation:

$$NaCNAgCN + NaCl + 2AgNO_3 = 2NaNO_3 + 2AgCN + AgCl.$$

Therefore the second minus the first reading represents the chlorin present in terms of silver nitrate.

SOAP.

61 MOISTURE[15].—OFFICIAL.

Weigh about 5 grams of the sample in a tared, 100 cc. beaker, in which is previously placed a ½ inch layer of recently ignited, dry sand and a small glass rod; if the soap is hard, cut it off in very thin strips. Add 25 cc. of alcohol, or more if necessary, and dissolve on the water bath, stirring constantly. Evaporate the alcohol, heat in an oven at 110°C. until the soap is nearly dry, and weigh, then dry again for 30 minutes and weigh. Continue this alternate drying and weighing until the weight changes only a few milligrams during the course of 30 minutes' drying.

62 POTASSIUM AND SODIUM[16].—OFFICIAL.

Dissolve about 5 grams of the soap in water; decompose with hydrochloric acid; filter off the water and wash the fat with cold water. Determine both potassium and sodium in the filtrate as directed under **II, 13**.

SODA LYE.

63 CARBONATE AND HYDROXID[17].—OFFICIAL.

Weigh about 10 grams of the sample from a weighing bottle, dissolve in carbon dioxid-free water and make up to a definite volume. Titrate an aliquot of this solution with N/2 hydrochloric acid, using methyl orange as an indicator, and note the total alkalinity thus found. Transfer an equal aliquot to a graduated flask and add enough barium chlorid solution to precipitate all the carbonate, avoiding any unnecessary excess. Dilute to the mark with carbon dioxid-free water, stopper, shake and set aside. When the liquid becomes clear, pipette off one-half and titrate with N/2 hydrochloric acid, using phenolphthalein as an indicator. The number of cc. of N/2 acid required for this titration. multiplied by 2, gives the number of cc. of N/2 acid required to neutralize the sodium hydroxid present in the orginal aliquot. The difference between this figure and the nunber of cc. of N/2 hydrochloric acid required for the total alkalinity represents the number of cc. of N/2 acid required to neutralize the sodium carbonate present in the aliquot. Calculate the percentages of sodium carbonate and hydroxid present in the sample.

TOBACCO AND TOBACCO EXTRACT.

NICOTIN.

Kissling Method.—Official.

64 REAGENTS.

(a) *Alcoholic sodium hydroxid solution.*—Dissolve 6 grams of sodium hydroxid in 40 cc. of water and 60 cc. of 90 per cent alcohol.

(b) *Dilute sodium hydroxid solution.*—Dissolve 2 grams of sodium hydroxid in water and dilute to 500 cc.

(c) *N/10 sulphuric acid.*—One cc. is equivalent to 16.22 mg. of nicotin.

(d) *Phenacetolin solution.*—Prepare a 0.5 per cent alcoholic solution.

(e) *Cochineal solution.*—Prepare as directed under **I, 16** (**j**).

65 DETERMINATION.

Weigh 5–6 grams of tobacco extract, or 20 grams of finely powdered tobacco which has been previously dried at 60°C., to permit powdering into a small beaker. Add 10 cc. of the alcoholic sodium hydroxid and follow, in the case of tobacco extract, with enough pure powdered calcium carbonate to form a moist but not lumpy mass. Mix thoroughly, transfer to a Soxhlet extractor and exhaust for about 5 hours with ether. Evaporate the ether at a low temperature, and take up the residue with 50 cc. of the dilute sodium hydroxid solution. Transfer this residue by means of water to a 500 cc. Kjeldahl flask, and distil with steam, passing the distillate through a condenser cooled by a rapidly flowing current of water. Use a 3-bend outflow tube, and, to prevent bumping and frothing, add a few pieces of pumice and a small piece of paraffin. Distil until all the nicotin has passed over, the distillate usually varying from 400 to 500 cc. When the distllation is completed, only about 15 cc. of the liquid should remain in the flask. Titrate the distillate with N/10 sulphuric acid, using the phenacetolin or cochineal solution as indicator.

Silicotungstic Acid Method[18].—Official.

66 REAGENTS.

(a) *Silicotungstic acid solution.*—Prepare a 12 per cent solution of the silicotungstic acid having the following formula: $4H_2O.SiO_2.12WO_3.22H_2O$. (There are several silicotungstic acids. The acids $4H_2O.SiO_2.10WO_3.3H_2O$ and $4H_2O.SiO_2.12WO_3.20H_2O$ do not give crystalline precipitates with nicotin and should not be used.)

(b) *Sodium or potassium hydroxid solution* (*1 to 2*).

(c) *Dilute hydrochloric acid* (*1 to 4*).

67 DETERMINATION.

Weigh such an amount of the preparation as will contain preferably between 0.1 and 1.0 gram of nicotin (if the sample contains very little nicotin, about 0.1 per cent, do not increase the amount to the point where it interferes with the distillation); wash with water into a 500 cc. round-bottomed distillation flask; add a little paraffin to prevent frothing, a few small pieces of pumice and a slight excess of the sodium or potassium hydroxid, using phenolphthalein as an indicator. Distil rapidly in a current of steam through a well-cooled condenser, connected by means of an adapter with a suitable flask containing 10 cc. of the dilute hydrochloric acid. When distillation is well under way, heat the distillation flask to reduce the volume of the liquid as far as practicable without bumping or undue separation of insoluble matter. Distil until a few cc. of the distillate show no cloud or opalescence when treated with a drop of the silicotungstic acid and a drop of the dilute hydrochloric acid. Confirm the alkalinity of the residue in the distillation flask with phenolphthalein solution. Make up the distillate, which may amount to 1000–1500 cc., to a convenient volume (the solution may be concentrated on the steam bath without loss of nicotin), mix well and pass through a large dry filter if not clear. Test a portion with methyl orange to assure its acidity. Pipette an aliquot, containing about 0.1 gram of nicotin, into a beaker (if the samples contain very small amounts of nicotin, an aliquot containing as little as 0.01 gram of nicotin may be used), add to each 100 cc. of liquid 3 cc. of the dilute hydrochloric acid, or more if the necessity is indicated by the test with methyl orange, and 1 cc. of the silicotungstic acid for each 0.01 gram of nicotin supposed to be present. Stir thoroughly and let stand overnight. Before filtering, stir the precipitate to see that it settles quickly and is in crystalline form; then filter on an ashless filter and wash with cold dilute hydrochloric acid (1 to 1000). Transfer the paper and precipitate to a weighed platinum crucible, dry carefully, and ignite until all carbon is destroyed. Finally heat over a Teclu or Meker burner for not more than 10 minutes. The weight of the residue, multiplied by 0.114, gives the weight of nicotin present in the aliquot.

FORMALDEHYDE SOLUTIONS.

FORMALDEHYDE.

Hydrogen Peroxid Method[19].—*Official.*

68 REAGENTS.

(a) *N/1 sulphuric acid.*

(b) *N/1 sodium hydroxid.*—One cc. is equivalent to 30.02 mg. of formaldehyde.

(c) *Hydrogen peroxid.*—An approximately 3 per cent solution. If the hydrogen peroxid solution is acid, neutralize with (b), using litmus solution as indicator.

(d) *Litmus solution.*—A solution of purified litmus.

69 DETERMINATION.

Measure 50 cc. of N/1 sodium hydroxid into a 500 cc. Erlenmeyer flask and add 50 cc. of the hydrogen peroxid. Then add 3 grams of the formaldehyde solution under examination, allowing the point of the pipette to reach nearly to the liquid in the flask. Place a funnel in the neck of the flask and heat on the steam bath for 5 minutes, shaking occasionally. Remove from the steam bath, wash the funnel with water, cool the flask to about room temperature and titrate the excess of N/1 sodium hydroxid with N/1 acid, using the litmus solution as indicator. It is necessary to cool the flask before titration with the acid to get a sharp end point with the litmus. From the amount of N/1 sodium hydroxid consumed and the weight of the sample calculate the per cent of formaldehyde.

Cyanid Method[20].—*Official.*

70 REAGENTS.

(a) *N/10 silver nitrate.*

(b) *N/10 ammonium thiocyanate.*

(c) *Potassium cyanid solution.*—Dissolve 3.1 grams of potassium cyanid in 500 cc. of water.

(d) *Dilute nitric acid (1 to 1).*

71 DETERMINATION.

Treat 15 cc. of the N/10 silver nitrate with 6 drops of the dilute nitric acid in a 50 cc. volumetric flask; add 10 cc. of the potassium cyanid solution, dilute to the mark, shake well, filter through a dry filter and titrate 25 cc. of the filtrate with N/10 ammonium thiocyanate as directed under **II, 17.** Acidify another 15 cc. portion of the N/10 silver nitrate with 6 drops of the dilute nitric acid and treat with 10 cc. of the potassium cyanid solution to which has been added a measured quantity (the weight of which must be calculated from the specific gravity) of the formaldehyde solution containing not over 2.5 grams of a 1 per cent solution or its equivalent. Make up to 50 cc., filter and titrate a 25 cc. aliquot with the N/10 ammonium thiocyanate for the excess of silver as before. The difference between the number of cc. of N/10 ammonium thiocyanate used in these 2 titrations, multiplied by 2, gives the number of cc. of N/10 ammonium thiocyanate corresponding to the potassium cyanid used by the formaldehyde. Calculate the per cent of formaldehyde present (1 cc. of N/10 ammonium thiocyanate is equivalent to 3 mg. of formaldehyde (HCHO)).

LIME-SULPHUR SOLUTIONS.

TOTAL SULPHUR[21].—OFFICIAL.

72 PREPARATION OF SAMPLE.

Weigh 10 grams of the solution, transfer to a 250 cc. graduated flask, and immediately dilute to the mark with recently boiled and cooled water. Mix thoroughly and transfer to a number of small bottles, filling them entirely and avoiding contact of the solution with air as much as possible. Stopper these bottles, seal with paraffin and preserve in a dark, cool place.

73 DETERMINATION.

(Sulphur-free reagents should be used for all work.)

Dissolve 2–3 grams of sodium peroxid in 50 cc. of cold water in a 250 cc. beaker. Transfer a 10 cc. aliquot of the solution prepared for analysis as directed under **72** to this aqueous solution of sodium peroxid, keeping the tip of the pipette constantly just under the surface of the liquid until necessary to raise it for drainage at the end. Use a clean dry pipette for measuring each portion. Cover the beaker with a watch glass and heat on the steam bath, with occasional stirring, until all the sulphur is oxidized to sulphate, which is shown by the disappearance of the yellow color. Wash off the watch glass and the sides of the beaker, acidify with hydrochloric acid[22], evaporate to complete dryness, treat with water acidified with hydrochloric acid, boil and filter to remove silica. Dilute the filtrate to 300 cc., add 50 cc. of concentrated hydrochloric acid, heat to boiling, and add 10 per cent barium chlorid solution slowly and with constant stirring. The barium chlorid should be added at such a rate that about 4 minutes are required for running in the necessary amount (11 cc. for 1 gram of barium sulphate). The rate is best regulated by attaching a suitable capillary tip to the burette containing the barium chlorid solution. Evaporate to dryness on the steam bath, take up with hot water, filter through a quantitative filter, wash until free from chlorids, ignite carefully and heat to constant weight over a Bunsen burner. Calculate the sulphur from the weight of barium sulphate, using the factor 0.1373.

74 MONOSULPHID EQUIVALENT[24].—TENTATIVE.

Dilute 10 cc. of the solution prepared as directed under **72** to about 30 cc. with recently boiled and cooled water and titrate with N/10 iodin solution until the yellow color just disappears. (There should be no difficulty in determining this end point, but if there is, a small crystal of sodium nitroprussid may be used. It must not be added until the end point is practically reached, since if the blue color is well developed it can not be destroyed except by an excess of iodin). From the number of cc. of N/10 iodin used, calculate the monosulphid equivalent as follows: cc. of N/10 iodin × 0.0016 = number of grams of sulphur as monosulphid equivalent.

THIOSULPHATE SULPHUR.

Zinc Chlorid Method[21].—*Official.*

75 REAGENT.

Ammoniacal zinc chlorid solution.—Dissolve 50 grams of pure zinc chlorid in about 500 cc. of water, add 125 cc. of ammonium hydroxid (sp. gr. 0.90) and 50 grams of ammonium chorid[23] and dilute to 1 liter.

76 DETERMINATION.

To 50 cc. of water in a 200 cc. graduated flask, add, in the manner indicated under **73**, 50 cc. of the solution prepared as directed under **72**. Add a slight excess of the ammonical zinc chlorid solution and dilute to the mark. Shake thoroughly and filter through a dry filter. To 100 cc. of the filtrate add a few drops of methyl orange or methyl red and exactly neutralize with N/10 hydrochloric acid. Titrate the neutral solution with approximately N/20 iodin solution, **3** (c), using a few drops of starch solution as indicator. From the number of cc. of iodin solution used, calculate the thiosulphate sulphur present. As the value of the iodin solution is given in terms of arsenious oxid (As_2O_3), it is necessary to multiply this value by 1.296 to obtain the equivalent of thiosulphate sulphur.

77 *Iodin Titration Method*[24].—*Tentative.*

Continue the titration of the solution used in the determination of the monosulphid equivalent, **74**, with N/10 iodin solution, letting the iodin act as its own indicator until a small drop produces a slight permanent coloration. From the number of cc. of N/10 iodin used, calculate the thiosulphate sulphur as follows: cc. of N/10 iodin × 0.0064 = number of grams of sulphur as thiosulphate.

SULPHID SULPHUR.

78 *Zinc Chlorid Method*[21].—*Official.*

To 10–15 cc. of water in a small beaker add, in the manner indicated under **73**, a 10 cc. aliquot of the solution prepared as directed in **72**. Calculate the amount of ammoniacal zinc chlorid solution (**75**) necessary to precipitate all the sulphur in the aliquot and add a slight excess. Stir thoroughly, then filter, wash the precipitate 3 or 4 times with cold water and transfer filter paper and precipitate to the beaker in which the precipitation was made. Cover with water, disintegrate the paper with a glass rod and add about 3 grams of sodium peroxid, keeping the beaker well covered with a watch glass. Warm on the steam bath with frequent shaking until all the sulphur is oxidized to sulphate, adding more sodium peroxid if necessary. Make slightly acid with hydrochloric acid, filter to remove shreds of filter paper, wash thoroughly with hot water and determine the sulphur in the filtrate as directed under **73**.

79 *Iodin Titration Method*[24].—*Tentative.*

Allow the solution from **77** to stand several hours with occasional stirring, or acidify with a few drops of dilute hydrochloric acid and warm gently with stirring, filter and wash thoroughly with warm water. Place the filter paper with the sulphur in a small vessel and dissolve the sulphur in about 15 cc. of sodium hydroxid solution (1 to 3) by heating gently on a steam or water bath for 1–1.5 hours (do not boil). Keep the flask covered and shake gently a few times during the digestion to remove the sulphur from the sides. Oxidize by adding 2–3 grams of sodium peroxid dissolved in 50 cc. of cold water and complete the determination as directed under **73**, beginning with "Cover the beaker with a watch glass".

80 *Indirect Method.—Tentative.*

The difference between the total sulphur and the sum of the thiosulphate sulphur and sulphate sulphur is the sulphid sulphur.

81 SULPHATE SULPHUR.—OFFICIAL.

To the solution from the determination of thiosulphate sulphur, **76**, add 2 or 3 drops of hydrochloric acid, precipitate in the cold with 10 per cent barium chlorid solution, allow to stand overnight, filter, calculate the sulphur from the weight of barium sulphate and report as sulphate sulphur.

82 *Iodin Titration Method*[24].—*Tentative.*

To the filtrate from the determination of thiosulphate sulphur, add several drops of hydrochloric acid, precipitate in the cold with 5 cc. of 10 per cent barium chlorid solution, allow to stand overnight, filter, calculate the sulphur from the weight of barium sulphate and report as sulphate sulphur.

83 TOTAL LIME.—OFFICIAL.

To 25 cc. of the solution, prepared as directed under **72**, add 10 cc. of concentrated hydrochloric acid, evaporate to dryness on the steam bath, treat with water and a little hydrochloric acid, warm until all the calcium chlorid is dissolved, and filter to remove sulphur and any silica that may be present. Dilute the filtrate to a bulk of 200–250 cc., heat to boiling, add a few cc. of ammonia in excess, and then an excess of saturated solution of ammonium oxalate. Continue the boiling until the precipitated calcium oxalate assumes a well defined granular form, allow to stand for an hour, filter and wash a few times with hot water. Ignite in a platinum crucible over a blast lamp to constant weight and weigh as calcium oxid.

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VII. FOODS AND FEEDING STUFFS.—GENERAL METHODS.

1

PREPARATION OF SAMPLE.—OFFICIAL.

Grind the sample so that it will pass through a sieve having circular openings $\frac{1}{25}$ inch (1 mm.) in diameter. If the sample can not be ground, reduce it to as fine a state as possible.

MOISTURE.

2

Drying with Heat.—Official.

Dry a quantity of the substance, representing about 2 grams of dry material, in a current of dry hydrogen or in vacuo at the temperature of boiling water to constant weight (approximately 5 hours). If the substance be contained in a glass vessel, the latter should not be in contact with the boiling water.

3

Drying in Vacuo without Heat.—Official.

Mix the sample thoroughly and weigh by difference 2-5 gram portions from a stoppered weighing bottle into tared, covered crucibles. Where subsequent fat determinations are to be made, fat extraction cones may be used. Substances that dry down to horn-like material should be mixed with fat-free cotton or other suitable material (previously tared with the container). Place 200 cc. of fresh concentrated sulphuric acid in a strong, tight 6 inch vacuum desiccator. Put triplicate samples in separate desiccators, and exhaust by means of a vacuum pump. If a pump is not available, place 10 cc. of ether in a small beaker in the desiccator and exhaust with a water filter pump.

Between the pump and the desiccator interpose an empty bottle, next to the desiccator, and a bottle of water. Draw the air from the desiccator through the water and turn the desiccator stop-cock at just the instant when the water begins to rise in the tube leading from the empty bottle.

Gently rotate the desiccator 4 or 5 times during the first 12 hours to mix the sulphuric acid with the water which has collected as an upper layer. At the end of 24 hours open the desiccator, causing the incoming air to bubble through concentrated sulphuric acid, and make the first weighing. After weighing place in a desiccator containing fresh concentrated sulphuric acid and exhaust as before. Rotate the desiccator several times during the interval and weigh again after a suitable period of drying. Repeat this process of drying in vacuo over sulphuric acid until the weight is constant.

4

ASH.—OFFICIAL.

Char a quantity of the substance, representing about 2 grams of the dry material, and burn until free from carbon at a low heat, not to exceed dull redness. If a carbon-free ash can not be obtained in this manner, exhaust the charred mass with hot water, collect the insoluble residue on a filter, burn till the ash is white or nearly so, and then add the filtrate to the ash and evaporate to dryness. Heat to low redness till the ash is white or grayish white and weigh.

5

CRUDE PROTEIN.—OFFICIAL.

Determine nitrogen as directed under **I, 18, 21** or **23**, and multiply the result by 6.25.

ALBUMINOID NITROGEN.—OFFICIAL.

6

REAGENT.

Cupric hydroxid.—Dissolve 100 grams of pure copper sulphate in 5 liters of water, add 2.5 cc. of glycerol, and then dilute sodium hydroxid solution until the liquid is just alkaline; filter, rub the precipitate up with water containing 5 cc. of glycerol per liter and wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent of glycerol, thus preparing a uniform gelatinous mass that can be measured with a pipette. Determine the quantity of copper hydroxid per cc. of this mixture.

7

DETERMINATION.

Place 0.7 gram of the substance in a beaker, add 100 cc. of water, and heat to boiling; or, in case of substances rich in starch, heat on the water bath for 10 minutes; add a quantity of the reagent, prepared as directed in **6**, containing about 0.5 gram of the hydroxid; stir thoroughly, filter when cold, wash with cold water, and, without removing the precipitate from the filter, determine the nitrogen as directed under **I**, **18**, **21** or **23**, adding sufficient potassium sulphid solution to completely precipitate all of the copper and mercury. The filter paper used must be practically free from nitrogen. If the material (such as seeds, seed residue, or oil cake) is rich in alkaline phosphates, add, to decompose the alkaline phosphates, 1–2 cc. of a concentrated potash or soda alum solution, free from ammonia, then the copper hydroxid, and mix well by stirring. If this is not done, copper phosphate and free alkali may be formed, and the protein-copper precipitate partially dissolved in the alkaline liquid.

8

AMIDO NITROGEN.—OFFICIAL.

Subtract the amount of albuminoid nitrogen from the amount of total nitrogen to obtain the amido nitrogen.

CRUDE FAT OR ETHER EXTRACT.

Direct Method.—Official.

9

REAGENT.

Anhydrous ether.—Wash any of the commercial brands of ether with 2 or 3 successive portions of water, add solid sodium or potassium hydroxid, and let stand until most of the water has been abstracted from the ether. Decant into a dry bottle, add small pieces of carefully cleaned metallic sodium, and let stand until there is no further evolution of hydrogen gas. Keep the ether, thus dehydrated, over metallic sodium in lightly stoppered bottles.

10

DETERMINATION.

Large quantities of soluble carbohydrates may interfere with the complete extraction of the fat. In such cases extract with water before proceeding with the determination. Extract about 2 grams of material, dried as under **2** or **3**, with the anhydrous ether for 16 hours. Dry the extract at the temperature of boiling water for 30 minutes, cool in a desiccator, and weigh; continue, at 30 minute intervals, this alternate drying and weighing to constant weight. For most feeds a period of 1–1.5 hours is required.

11

Indirect Method.—Official.

Determine the moisture, as directed in **2** or **3**, then extract the dried substance for 16 hours as directed under **10**, dry again and regard the loss of weight as ether extract.

SUCROSE.

OPTICAL METHODS.

12 GENERAL DIRECTIONS FOR RAW SUGARS.—OFFICIAL.

(Rules[1] of the International Commission for Unifying Methods of Sugar Analysis.)

"In general all polarizations are to be made at 20°C.

"The verification of the saccharimeter must also be made at 20°C. For instruments using the Ventzke scale 26 grams of pure dry sucrose, weighed in air with brass weights, dissolved in 100 metric cc. at 20°C. and polarized in a room, the temperature of which is also 20°C., must give a saccharimeter reading of exactly 100.00. The temperature of the sugar solution during polarization must be kept constant at 20°C.

"For countries where the mean temperature is higher than 20°C., saccharimeters may be adjusted at 30°C. or any other suitable temperature, under the conditions specified above, provided that the sugar solution be made up to volume and polarized at this same temperature.

"In effecting the polarization of substances containing sugar employ only half-shade instruments." The saccharimeter used can be either single or double wedge and should be a half-shadow instrument with either double or triple field.

"During the observation keep the apparatus in a fixed position and so far removed from the source of light that the polarizing Nicol is not warmed.

"As sources of light employ lamps which give a strong illumination such as triple gas burner with metallic cylinder, lens and reflector; gas lamps with Auer (Welsbach) burner; electric lamp; petroleum duplex lamp; sodium light." Whenever there is any irregularity in the sources of light such as that due to the convolutions of the filament in the case of electric light or to the meshes of the gauze in the case of the Welsbach light, place a thin ground-glass plate between the source of light and the polariscope so as to render the illumination uniform.

"Before and after each set of observations the chemist must satisfy himself of the correct adjustment of his saccharimeter by means of standardized quartz plates. He must also previously satisfy himself of the accuracy of his weights, polarization flasks, observation tubes and cover-glasses. (Scratched cover-glasses must not be used.) Make several readings and take the mean thereof, but no one reading may be neglected." Such plates are standardized to read to the second decimal point and by their use a quick and at the same time accurate test can be made. In using such plates for testing saccharimeters, it is necessary that the instrument, as well as the plate, be at 20°C. before making a reading. Different points of the scale, preferably 20°, 50°, 80°, and 100°, (sugar scale) should be tested against the plates.

"In making a polarization use the whole normal weight for 100 cc. or a multiple thereof for any corresponding volume.

"As clarifying and decolorizing agents use either basic acetate of lead, alumina cream, or concentrated solution of alum. Boneblack and decolorizing powders are to be excluded." Whenever reducing sugars are determined in the solution for polarizing, use only neutral lead acetate for clarification as basic lead acetate causes precipitation of some of the reducing sugars. In addition to these clarifying agents, neutral lead acetate and basic lead nitrate have been made official by the Association.

"After bringing the solution exactly to the mark at the proper temperature, and after wiping out the neck of the flask with filter paper, pour all of the well-shaken clarified sugar solution on a rapidly acting filter. Reject the first portions of the filtrate, and use the rest, which must be perfectly clear, for polarization." It is advisable to reject the first 20 cc. that run through, then cover the funnel with a watch glass and use the remainder for polarization. In no case should the whole solution or any part be returned to the filter. If cloudy after the 20 cc. have been rejected, begin a new determination.

"Whenever white light is used in polarimetric determinations, the same must be filtered through a solution of potassium dichromate of such a concentration that the percentage content of the solution multiplied by the length of the column of the solution in centimeters is equal to nine." This concentration must be doubled in reading carbohydrate materials of high rotation dispersion, such as commercial glucose, etc.

13 PREPARATION AND USE OF CLARIFYING REAGENTS.—OFFICIAL.

(a) *Basic lead acetate solution.*—Boil 430 grams of neutral lead acetate, 130 grams of litharge, and 1 liter of water for 30 minutes. Allow the mixture to cool and settle and dilute the supernatant liquid to a specific gravity of 1.25 with recently boiled water. Solid basic lead acetate may be substituted for the normal salt and litharge in the preparation of the solution.

(b) *Alumina cream.*—Prepare a cold saturated solution of alum in water. Add ammonium hydroxid with constant stirring until the solution is alkaline to litmus, allow the precipitate to settle and wash by decantation with water until the wash water gives only a slight test for sulphates with barium chlorid solution. Pour off the excess of water and store the residual cream in a stoppered bottle.

(c) *Dry basic lead acetate.*—This clarifying agent is obtained as a dry powdered salt and should contain 72.8 per cent of lead, which corresponds to a composition of $3Pb(C_2H_3O_2)_2.2PbO$. Dissolve the normal or half-normal weight of the sugar solution in a sugar flask with water and complete the volume. Add a small quantity of the dry salt and shake, then add more and shake again, repeating until completely precipitated but avoiding any excess. Of this salt 0.1346 gram is equivalent to 1 cc. of the basic lead acetate solution, described under (a). When molasses or any other substance producing a heavy precipitate is being clarified, some dry, coarse sand should be added to break up the balls of basic lead acetate and the precipitate. (This method is to have equal weight with the use of a solution of basic lead acetate in clarifying cane, sorghum, and beet products.)

(d) *Neutral lead acetate.*—Prepare a saturated solution of neutral lead acetate and add it to the sugar solution before completing to volume. Its use is imperative when determining the reducing sugars in the solution used for polarization.

(e) *Basic lead nitrate.*—(1) Dissolve 250 grams of lead nitrate in water and make up to 500 cc. (2) Dissolve 25 grams of sodium hydroxid in water and make up to 500 cc.

Add equal amounts of (1) and (2) to the sugar solution, shake, and add more if complete precipitation has not occurred, but avoid an excess. Then complete the volume with water. When this solution is used for clarification, the factor in the Clerget determination becomes 143.5 instead of 142.66.

DETERMINATION OF SUCROSE IN THE ABSENCE OF RAFFINOSE.

(In the presence of much levulose, as in honeys and fruits products, the optical method for sucrose gives too high results.)

14 *By Polarization Before and After Inversion with Hydrochloric Acid.—Official.*

Dissolve the normal weight (26 grams) of the substance in water, add basic lead acetate carefully, avoiding any excess, then 1–2 cc. of alumina cream, shake, and dilute to 100 cc., filter, rejecting the first 20 cc. of the filtrate, cover the funnel with a watch glass and, when sufficient filtrate is collected, polarize in a 200 mm. tube. The reading so obtained is the direct reading (P of formula given below) or polarization before inversion. For the invert reading, remove the lead from the solution either (1) by adding anhydrous potassium oxalate, a little at a time, to the remaining solution, avoiding an excess and removing the precipitated lead by filtration; or, (2) by adding anhydrous sodium carbonate under the same conditions. Introduce 50 cc. of the lead-free filtrate into a 100 cc. flask (if sodium carbonate was used for removing the lead, neutralize carefully the excess of sodium carbonate with a few drops of dilute hydrochloric acid) and add 25 cc. of water. Then add, little by little, while rotating the flask, 5 cc. of hydrochloric acid (sp. gr. 1.20). Heat the flask after mixing, in a water bath kept at 70°C. The temperature of the solution in the flask should reach 67°–69°C. in 2.5–3 minutes. Maintain a temperature of as nearly 69°C. as possible for 7–7.5 minutes, making the total time of heating 10 minutes. Remove the flask and cool the contents rapidly to 20°C. and dilute to 100 cc. Polarize this solution in a tube provided with a lateral branch and a water jacket, maintaining a temperature of 20°C. This reading must be multiplied by 2 to obtain the invert reading. If it is necessary to work at a temperature other than 20°C., which is allowable within narrow limits, the volumes must be completed and both direct and invert polarizations must be made at exactly the same temperature.

The inversion may also be accomplished as follows: (1) To 50 cc. of the clarified solution, freed from lead, add 5 cc. of hydrochloric acid (sp. gr. 1.20) and set aside for 24 hours at a temperature not below 20°C.; or, (2) if the temperature be above 25°C. set aside for 10 hours. Make up to 100 cc. at 20°C. and polarize as directed above.

Calculate sucrose by one of the following formulas:

For substances in which the invert solution contains more than 12 grams of invert sugar per 100 cc.—The following formula is to be used when substances like raw sugars are polarized:

$$S = \frac{100\,(P - I)}{142.66 - \frac{T}{2}}\text{, in which}$$

S = per cent of sucrose;
P = direct reading, normal solution;
I = invert reading, normal solution;
T = temperature at which readings are made.

For substances in which the concentration of the invert solution is less than 12 grams per 100 cc.—The following formula, which takes into account the concentration of the sugar in solution, should be used in all other cases:

$$S = \frac{100\,(P-I)}{142.66 - \frac{T}{2} - 0.0065\left[142.66 - \frac{T}{2} - (P-I)\right]}\text{ in which}$$

S = per cent of sucrose;
P = direct reading, normal solution;
I = invert reading, normal solution;
T = temperature.

By Polarization Before and After Inversion with Invertase.—Official.

15 REAGENT.

Invertase solution[2].—Mix 1 kilo of pressed baker's or brewer's yeast with 1 liter of tap water and 50 cc. of toluene and keep at room temperature 2–3 days to allow autolysis to proceed to the stage of maximum inverting activity. Then add neutral lead acetate in slight excess, filter, precipitate the lead in the filtrate with hydrogen sulphid, filter again and then dialyze the filtrate thoroughly in a collodion sac. Preserve in an ice box the dialyzed solution with the addition of a little toluene to prevent the growth of micro-organisms. Note the optical activity of the invertase solution and correct the invert reading according to the amount of the solution used.

16 DETERMINATION.

Dissolve the normal weight (26 grams) of the substance in water, clarify, make up to volume, and take the direct polarization (P) as directed under **14**. If lead has been used as a clarifying agent, remove the excess of lead from the filtrate with anhydrous sodium carbonate or potassium oxalate and filter. To 50 cc. of the filtrate in a 100 cc. flask add acetic acid, drop by drop, until the reaction is acid to litmus, add 10 cc. of the invertase solution, fill the flask with water nearly to 100 cc. and let stand in a warm place (about 40°C.) overnight. Cool and make up to 100 cc. at 20°C. Polarize at 20°C. in a 200 mm. tube. Allow the solution to remain in the tube for an hour and repeat the polarization. If there is no change from the previous reading, the inversion is complete, whereupon the reading and temperature of the solution are carefully noted. Correct the reading for the optical activity of the invertase solution and then multiply by 2. Calculate the percentage of sucrose by the following formula:

$$S = \frac{100\,(P-I)}{142 - \frac{T}{2} - 0.0065\left[142 - \frac{T}{2} - (P-I)\right]} \text{ in which}$$

S = per cent of sucrose;
P = direct reading;
I = invert reading;
T = temperature at which invert reading is made.

17 DETERMINATION OF SUCROSE AND RAFFINOSE.—OFFICIAL.

(Of value chiefly in the analysis of beet products.)

If the direct reading is more than 1° higher than the per cent of sucrose as calculated by the formula given under **14**, raffinose is probably present. Calculate sucrose and raffinose by the following formula:

$$S = \frac{0.5124\,P - I}{0.839}; \quad R = \frac{P - S}{1.852} \text{ in which}$$

P = direct reading, normal solution;
I = invert reading, normal solution;
S = per cent of sucrose;
R = per cent of anhydrous raffinose.

The above formula assumes that the polarizations are made at exactly 20°C. If the temperature (T) is other than 20°C., the following formula should be used:

$$S = \frac{P\,(0.4724 + 0.002\,T) - I}{0.899 - 0.003\,T}$$

Having calculated S, then $R = \frac{P - S}{1.852}$

CHEMICAL METHODS.

18 DETERMINATION OF SUCROSE FROM REDUCING SUGARS BEFORE AND AFTER INVERSION.—OFFICIAL.

Determine the reducing sugars (clarification having been effected with *neutral* lead acetate, never with basic lead acetate) as directed under **25** and calculate to invert sugar from **XXX**, Table 1. Invert the solution as directed under **14** or **16**, exactly neutralize the acid, and again determine the reducing sugars, but calculate them to invert sugar from the same table as referred to above, using the invert sugar column alone. Deduct the percentage of invert sugar obtained before inversion from that obtained after inversion, and multiply the difference by 0.95, the result being the per cent of sucrose. The solutions should be diluted in both determinations so that not more than 240 mg. of invert sugar are present in the amount taken for reduction. It is important that all lead be removed from the solution with potassium oxalate before reduction.

REDUCING SUGARS.

INVERT SUGAR.

Approximate Volumetric Method for Rapid Work.—Tentative.

19 REAGENT.

Soxhlet's Modification of Fehling's Solution.—Prepared by mixing, immediately before use, equal volumes of (**a**) and (**b**).

(**a**) *Copper sulphate solution.*—Dissolve 34.639 grams of copper sulphate ($CuSO_4.5H_2O$) in water, dilute to 500 cc. and filter through prepared asbestos.

(**b**) *Alkaline tartrate solution.*—Dissolve 173 grams of Rochelle salts and 50 grams of sodium hydroxid in water, dilute to 500 cc., allow to stand for 2 days and filter through prepared asbestos.

20 STANDARDIZATION OF COPPER SOLUTION.

Since the factor of calculation varies with the minute details of manipulation, every operator must determine a factor for himself, using a known solution of the pure sugar that he desires to determine, and keeping the conditions the same as those used for the determination.

Standardize the solution for invert sugar in the following manner:

Dissolve 4.75 grams of pure sucrose in 75 cc. of water, add 5 cc. of hydrochloric acid (sp. gr. 1.20) and invert as directed under **14**. Neutralize the acid with sodium hydroxid solution and dilute to 1 liter. Ten cc. of this solution contain 0.050 gram of invert sugar, which should reduce 10 cc. of the reagent. The strength of the copper solution should never be taken as a constant, but should be checked against the sugar.

21 DETERMINATION.

Place 10 cc. of the reagent in a large test tube and add 10 cc. of water. Heat to boiling, and add gradually small portions of the solution of the material to be tested until the copper has been completely reduced, boiling after each addition to complete the reaction. Two minutes' boiling is required for complete reduction when the full amount of sugar solution has been added in one portion. When the end is nearly reached and the amount of sugar solution to be added can no longer be judged by the color of the solution, remove a small portion of the liquid and filter rapidly into a small porcelain crucible or on a test plate; acidify with dilute acetic acid and test for copper with dilute potassium ferrocyanid solution. The sugar solution should be of such strength as to give a burette reading of 15–20 cc., and the number of successive additions should be as small as possible.

Soxhlet Volumetric Method.—Tentative.

22

REAGENT.

The reagent used is described under **19**.

23

DETERMINATION.

Make a preliminary titration to determine the approximate percentage of reducing sugar in the material under examination. Prepare a solution which contains approximately 1 per cent of reducing sugar. Place in a beaker 100 cc. of the reagent and approximately the amount of the sugar solution for its complete reduction. Boil for 2 minutes. Filter through a folded filter and test a portion of the filtrate for copper by use of dilute acetic acid and dilute potassium ferrocyanid solution. Repeat, varying the volume of sugar solution, until 2 successive amounts are found which differ by 0.1 cc., one giving complete reduction and the other leaving a small amount of copper in solution. The mean of these 2 readings is taken as the volume of the solution required for the complete precipitation of 100 cc. of the reagent.

Under these conditions 100 cc. of the reagent require 0.494 gram of invert sugar for complete reduction. Calculate the percentage by the following formula:

$$\text{Per cent of invert sugar} = \frac{100 \times 0.494}{VW};$$

V = the volume of the sugar solution required for the complete reduction of 100 cc. of the reagent,

W = the weight of the sample in 1 cc. of the sugar solution.

Munson and Walker General Method[3]*.—Official.*

24

REAGENTS.

(**a**) *Asbestos.*—Digest the asbestos, which should be the amphibole variety, with dilute hydrochloric acid (1 to 3) for 2–3 days. Wash free from acid, digest for a similar period with 10 per cent sodium hydroxid solution, and then treat for a few hours with hot alkaline tartrate solution (old alkaline tartrate solutions that have stood for some time may be used for this purpose) of the strength employed in sugar determinations. Then wash the asbestos free from alkali, digest for several hours with dilute nitric acid (1 to 3) and, after washing free from acid, shake with water into a fine pulp. In preparing the Gooch crucible, make a film of asbestos ¼ inch thick and wash thoroughly with water to remove fine particles of asbestos. If the precipitated cuprous oxid is to be weighed as such, wash the crucible with 10 cc. of alcohol, then with 10 cc. of ether, dry for 30 minutes at 100°C., cool in a desiccator and weigh.

(**b**) The solutions used are described under **19**.

25

PRECIPITATION OF CUPROUS OXID.

Transfer 25 cc. each of the copper sulphate and alkaline tartrate solutions to a 400 cc. beaker of alkali-resistant glass and add 50 cc. of reducing sugar solution, or, if a smaller volume of sugar solution is used, add water to make the final volume 100 cc. Heat the beaker upon an asbestos gauze over a Bunsen burner, regulate the flame so that boiling begins in 4 minutes and continue the boiling for exactly 2 minutes. (It is important that these directions be strictly observed and, in order to regulate the burner for this purpose, it is advisable to make preliminary tests, using 50 cc. of the reagent and 50 cc. of water before proceeding with the actual determination.) Keep the beaker covered with a watch glass during the heating. Filter the cuprous oxid at once on an asbestos mat in a porcelain Gooch crucible, using suction. Wash the cuprous oxid thoroughly with water at a temperature of about 60°C., and either weigh directly as cuprous oxid as in **26**, or determine the amount of reduced copper by one of the methods under **28–33**, respectively. Conduct a blank determination, using 50 cc. of the reagent and 50 cc. of water, and, if the weight of cuprous oxid obtained exceeds 0.5 mg., correct the result of the reducing sugar determination accordingly. The alkaline tartrate solution deteriorates on standing and the amount of cuprous oxid obtained in the blank increases.

DETERMINATION OF REDUCED COPPER.

26 *I. Direct Weighing of Cuprous Oxid.—Official.*

Prepare a Gooch crucible as directed under **24** (**a**).

Collect the precipitated cuprous oxid on the mat, as directed under **25**, wash thoroughly with hot water, then with 10 cc. of alcohol, and finally with 10 cc. of ether. Dry the precipitate for 30 minutes in a water oven at the temperature of boiling water; cool and weigh. Calculate the weight of metallic copper, using the factor 0.8882. Obtain from **XXX**, Table 1, the weight of invert sugar equivalent to the weight of copper found.

This method should be used only for determinations in pure sugar solutions. In all other products the copper of the cuprous oxid should be determined by one of the following methods, since the cuprous oxid is very apt to be contaminated with foreign matter.

The number of milligrams of copper reduced by a given amount of reducing sugar differs when sucrose is present and when it is absent. In the tables the absence of sucrose is assumed except in the two columns under invert sugar, where one for mixtures of invert sugar and sucrose containing 0.4 gram of total sugar in 50 cc. of solution, and one for invert sugar and sucrose when the 50 cc. of solution contains 2 grams of total sugar are given, in addition to the column for invert sugar alone.

Volumetric Thiosulphate Method[4]*.—Official.*

27 REAGENT.

Standard thiosulphate solution.—Prepare a solution of sodium thiosulphate containing 19 grams of pure crystals in 1 liter. Weigh accurately about 0.2 gram of pure copper foil and place in a flask of 250 cc. capacity. Dissolve by warming with 5 cc. of a mixture of equal volumes of strong nitric acid and water. Dilute to 50 cc., boil to expel the red fumes, add 5 cc. of strong bromin water, and boil until the bromin is completely driven off. Remove from the heat and add a slight excess of strong ammonium hydroxid (about 7 cc. is required). Again boil until the excess of ammonia is expelled, as shown by a change of color of the liquid, and a partial precipitation. Then add a slight excess of strong acetic acid (3–4 cc. of 80 per cent acid) and boil for a minute. Cool to room temperature and add 10 cc. of 30 per cent potassium iodid solution. Titrate at once with the thiosulphate solution until the brown tinge has become weak, then add sufficient starch indicator [**VI, 3** (**a**)] to produce a marked blue coloration. Continue the titration cautiously until the color due to free iodin has entirely vanished. The blue color changes toward the end to a faint lilac. If at this point the thiosulphate be added drop by drop and a little time allowed for complete reaction after each addition, there is no difficulty in determining the end point within a single drop. One cc. of the thiosulphate solution will be found to correspond to about 0.005 gram of copper.

28 DETERMINATION.

After washing the precipitated cuprous oxid, cover the Gooch with a watch glass and dissolve the oxid by means of 5 cc. of warm nitric acid (1 to 1) poured under the watch glass with a pipette. Catch the filtrate in a 250 cc. flask, wash the watch glass and Gooch free of copper, using about 50 cc. of water. Boil to expel red fumes, add 5 cc. of bromin water, boil off the bromin and proceed exactly as directed under **27**.

29 *III. Volumetric Permanganate Method.—Official.*

Filter and wash the cuprous oxid as directed under **25**. Transfer the asbestos film to the beaker, add about 30 cc. of hot water, and beat the precipitate and asbestos thoroughly. Rinse the crucible with 50 cc. of a hot saturated solution of ferric sulphate in 20 per cent sulphuric acid, receiving the rinsings in the beaker containing the precipitate. After the cuprous oxid is dissolved, wash the solution into a large Erlenmeyer flask and immediately titrate with a standard solution of potassium permanganate, 1 cc. of which should be equivalent to 0.010 gram of copper. Standardize this solution by making 6 or more determinations with the same sugar solution, titrating one-half of the precipitates obtained, and determining the copper in the others by electrolysis. The average weight of copper obtained by electrolysis, divided by the average number of cc. of permanganate solution required for the titrations, gives the weight of copper equivalent to 1 cc. of the standard permanganate solution. A solution standardized with iron or oxalic acid will give too low a result.

30 *IV. Electrolytic Deposition from Sulphuric Acid Solution.—Official.*

Filter the cuprous oxid in a Gooch, wash the beaker and the precipitate thoroughly with hot water without transferring the precipitate to the filter. Wash the asbestos film and the adhering cuprous oxid into the beaker by means of hot dilute nitric acid. After the copper is all in solution, refilter through a thin film of asbestos in a Gooch and wash thoroughly with hot water. Add 10 cc. of sulphuric acid (1 to 4), and evaporate the filtrate on the steam bath until the copper salt has largely crystallized. Heat carefully on a hot plate or over asbestos until the evolution of white fumes shows that the excess of nitric acid is removed. Add 8–10 drops of nitric acid (sp. gr. 1.42) and rinse into a 100–125 cc. platinum dish. Deposit the copper by electrolysis. Wash thoroughly with water, then break the current, wash with alcohol and ether successively, dry at about 50°C., and weigh. If preferred, the electrolysis can be conducted in a beaker, the copper being deposited upon a weighed platinum electrode.

31 *V. Electrolytic Deposition from Sulphuric and Nitric Acid Solution.—Official.*

Filter and wash as directed under **30**. Transfer the asbestos film from the crucible to the beaker by means of a glass rod and rinse the crucible with about 30 cc. of a boiling mixture of dilute sulphuric and nitric acids, containing 65 cc. of sulphuric acid (sp. gr. 1.84) and 50 cc. of nitric acid (sp. gr. 1.42) per liter. Heat and agitate until solution is complete; filter and electrolyze as directed under **30**.

32 *VI. Electrolytic Deposition from Nitric Acid Solution.—Official.*

Filter and wash as directed under **30**. Transfer the asbestos film and adhering oxid to the beaker. Dissolve the oxid still remaining in the crucible by means of 2 cc. of nitric acid (sp. gr. 1.42), adding it with a pipette and receiving the solution in the beaker containing the asbestos film. Rinse the crucible with a jet of water, allowing the rinsings to flow into the beaker. Heat the contents of the beaker until the copper is all in solution, filter, dilute the filtrate to a volume of 100 cc. or more, and electrolyze. When a nitrate solution is electrolyzed, the first washing of the deposit should be made with water acidulated with sulphuric acid, in order to remove all the nitric acid before the current is interrupted.

33 VII. *Reduction in Hydrogen.—Official.*

Deposit an asbestos film on a perforated platinum disc or cone contained in a hard glass filtering tube, wash free from loose fibers, dry and weigh. Through this tube, previously moistened, filter the cuprous oxid immediately, using suction. Transfer the cuprous oxid to the tube through a removable funnel, and wash thoroughly with hot water, alcohol and ether successively. After drying, connect the tube with a supply of dry hydrogen, heat gently until the cuprous oxid is completely reduced to metallic copper, cool in the current of hydrogen, and weigh. If preferred, a Gooch crucible may be used for the filtration.

Herzfeld Gravimetric Method.—Official.

Method I.

(For materials containing 1.5% or less of invert sugar and 98.5% or more of sucrose.)

34 REAGENTS.

The reagents and solutions used are described under **24.**

35 DETERMINATION.

Prepare the solution of the material to be examined so as to contain 20 grams in 100 cc., free from suspended impurities by filtration and from soluble impurities by neutral lead acetate, removing the excess of lead by means of sodium carbonate. Place 50 cc. of the reagent and 50 cc. of the sugar solution in a 250 cc. beaker. Heat this mixture at such a rate that approximately 4 minutes are required to bring it to the boiling point, and boil for exactly 2 minutes. Add 100 cc. of cold recently boiled water. Filter immediately through asbestos, and determine the copper by one of the methods under **26**, **28–33**, respectively. Obtain the corresponding percentage of invert sugar from **36.**

36 TABLE 1.—HERZFELD TABLE[5].

For the determination of invert sugar in materials containing 1.5% or less of invert sugar and 98.5% or more of sucrose.

COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR	COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR	COPPER REDUCED BY 10 GRAMS OF MATERIAL	INVERT SUGAR
mg.	*per cent*	*mg.*	*per cent*	*mg.*	*per cent*
50	0.05	140	0.51	230	1.02
55	0.07	145	0.53	235	1.05
60	0.09	150	0.56	240	1.07
65	0.11	155	0.59	245	1.10
70	0.14	160	0.62	250	1.13
75	0.16	165	0.65	255	1.16
80	0.19	170	0.68	260	1.18
85	0.21	175	0.71	265	1.21
90	0.24	180	0.74	270	1.24
95	0.27	185	0.76	275	1.27
100	0.30	190	0.79	280	1.30
105	0.32	195	0.82	285	1.33
110	0.35	200	0.85	290	1.36
115	0.38	205	0.88	295	1.38
120	0.40	210	0.90	300	1.41
125	0.43	215	0.93	305	1.44
130	0.45	220	0.96	310	1.47
135	0.48	225	0.99	315	1.50

Method II.

(For materials containing more than 1.5% of invert sugar and less than 98.5% of sucrose.)

37 REAGENTS.

Same as described under **24**.

38 DETERMINATION.

Prepare a solution of the material to be examined in such a manner that it contains 20 grams in 100 cc. after clarification and removal of the excess of lead. Prepare a series of solutions in large test-tubes by adding 1, 2, 3, 4, and 5 cc. of this solution to each tube successively. Add 5 cc. of the reagent to each, heat to boiling, boil 2 minutes, and filter. Note the volume of sugar solution which gives the filtrate lightest in tint, but still distinctly blue. Place 20 times this volume of the sugar solution in a 100 cc. flask, dilute to the mark, and mix well. Use 50 cc. of the solution for the determination, which is conducted as described under **35**. For the calculation of the result use the following formulas and table of factors of Meissl and Hiller:

Let Cu = the weight of copper obtained;
P = the polarization of the sample;
W = the weight of the sample in the 50 cc. of the solution used for the determination;
F = the factor obtained from the table for the conversion of copper to invert sugar;

Then $\frac{Cu}{2} = Z$, approximate weight of invert sugar;

$Z \times \frac{100}{W} = Y$, approximate per cent of invert sugar;

$\frac{100\,P}{P+Y} = R$, approximate per cent of sucrose in mixture of sugars;

$100 - R = I$, approximate per cent of invert sugar;

$\frac{CuF}{W} =$ per cent of invert sugar.

The factor F for calculating copper to invert sugar is then found from **39**.

39

TABLE 2.

Meissl and Hiller's[6] factors for determinations in materials in which, of the total sugars present, more than 1.5% is invert sugar, and less than 98.5% is sucrose.

RATIO OF SUCROSE TO INVERT SUGAR=R : I.	APPROXIMATE ABSOLUTE WEIGHT OF INVERT SUGAR (Z)						
	200 milligrams	175 milligrams	150 milligrams	125 milligrams	100 milligrams	75 milligrams	50 milligrams
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
0 : 100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10 : 90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20 : 80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30 : 70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40 : 60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50 : 50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60 : 40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70 : 30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80 : 20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90 : 10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91 : 9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92 : 8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93 : 7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94 : 6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95 : 5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

Example: The polarization of a sugar is 86.4 and 50 cc. of solution containing 3.256 grams of sample gave 0.290 gram of copper.

$$\frac{\text{Cu}}{2} = \frac{0.290}{2} = 0.145 = \text{Z}$$

$$\frac{\text{Z} \times 100}{\text{W}} = 0.145 \times \frac{100}{3.256} = 4.45 = \text{Y}$$

$$100 - \text{R} = 100 - 95.1 = \text{I} = 4.9$$

$$\text{R} : \text{I} = 95.1 : 4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95 : 5 is nearest to the ratio of R to I, 95.1 : 4.9. Where these columns meet, we find the factor 51.2 which enters into the final calculation:

$$\frac{\text{Cu F}}{\text{W}} = \frac{0.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert sugar.}$$

In case there is no sucrose present, the following table may be used instead of the factors given in 39.

40 TABLE 3.—MEISSL'S TABLE[7].

For the determination of invert sugar alone.

[According to Wein.]

[Expressed in milligrams.]

COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR
90	46.9	135	70.8	180	95.2	225	120.4
91	47.4	136	71.3	181	95.7	226	120.9
92	47.9	137	71.9	182	96.2	227	121.5
93	48.4	138	72.4	183	96.8	228	122.1
94	48.9	139	72.9	184	97.3	229	122.6
95	49.5	140	73.5	185	97.8	230	123.2
96	50.0	141	74.0	186	98.4	231	123.8
97	50.5	142	74.5	187	99.0	232	124.3
98	51.1	143	75.1	188	99.5	233	124.9
99	51.6	144	75.6	189	100.1	234	125.5
100	52.1	145	76.1	190	100.6	235	126.0
101	52.7	146	76.7	191	101.2	236	126.6
102	53.2	147	77.2	192	101.7	237	127.2
103	53.7	148	77.8	193	102.3	238	127.8
104	54.3	149	78.3	194	102.9	239	128.3
105	54.8	150	78.9	195	103.4	240	128.9
106	55.3	151	79.4	196	104.0	241	129.5
107	55.9	152	80.0	197	104.6	242	130.0
108	56.4	153	80.5	198	105.1	243	130.6
109	56.9	154	81.0	199	105.7	244	131.2
110	57.5	155	81.6	200	106.3	245	131.8
111	58.0	156	82.1	201	106.8	246	132.3
112	58.5	157	82.7	202	107.4	247	132.9
113	59.1	158	83.2	203	107.9	248	133.5
114	59.6	159	83.8	204	108.5	249	134.1
115	60.1	160	84.3	205	109.1	250	134.6
116	60.7	161	84.8	206	109.6	251	135.2
117	61.2	162	85.4	207	110.2	252	135.8
118	61.7	163	85.9	208	110.8	253	136.3
119	62.3	164	86.5	209	111.3	254	136.9
120	62.8	165	87.0	210	111.9	255	137.5
121	63.3	166	87.6	211	112.5	256	138.1
122	63.9	167	88.1	212	113.0	257	138.6
123	64.4	168	88.6	213	113.6	258	139.2
124	64.9	169	89.2	214	114.2	259	139.8
125	65.5	170	89.7	215	114.7	260	140.4
126	66.0	171	90.3	216	115.3	261	140.9
127	66.5	172	90.8	217	115.8	262	141.5
128	67.1	173	91.4	218	116.4	263	142.1
129	67.6	174	91.9	219	117.0	264	142.7
130	68.1	175	92.4	220	117.5	265	143.2
131	68.7	176	93.0	221	118.1	266	143.8
132	69.2	177	93.5	222	118.7	267	144.4
133	69.7	178	94.1	223	119.2	268	144.9
134	70.3	179	94.6	224	119.8	269	145.5

40 TABLE 3.—MEISSL'S TABLE.—Concluded.

[Expressed in milligrams.]

COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR	COPPER	INVERT SUGAR
270	146.1	310	169.7	350	193.8	390	218.7
271	146.7	311	170.3	351	194.4	391	219.3
272	147.2	312	170.9	352	195.0	392	219.9
273	147.8	313	171.5	353	195.6	393	220.5
274	148.4	314	172.1	354	196.2	394	221.2
275	149.0	315	172.7	355	196.8	395	221.8
276	149.5	316	173.3	356	197.4	396	222.4
277	150.1	317	173.9	357	198.0	397	223.1
278	150.7	318	174.5	358	198.6	398	223.7
279	151.3	319	175.1	359	199.2	399	224.3
280	151.9	320	175.6	360	199.8	400	224.9
281	152.5	321	176.2	361	200.4	401	225.7
282	153.1	322	176.8	362	201.1	402	226.4
283	153.7	323	177.4	363	201.7	403	227.1
284	154.3	324	178.0	364	202.3	404	227.8
285	154.9	325	178.6	365	203.0	405	228.6
286	155.5	326	179.2	366	203.6	406	229.3
287	156.1	327	179.8	367	204.2	407	230.0
288	156.7	328	180.4	368	204.8	408	230.7
289	157.2	329	181.0	369	205.5	409	231.4
290	157.8	330	181.6	370	206.1	410	232.1
291	158.4	331	182.2	371	206.7	411	232.8
292	159.0	332	182.8	372	207.3	412	233.5
293	159.6	333	183.5	373	208.0	413	234.3
294	160.2	334	184.1	374	208.6	414	235.0
295	160.8	335	184.7	375	209.2	415	235.7
296	161.4	336	185.4	376	209.9	416	236.4
297	162.0	337	186.0	377	210.5	417	237.1
298	162.6	338	186.6	378	211.1	418	237.8
299	163.2	339	187.2	379	211.7	419	238.5
300	163.8	340	187.8	380	212.4	420	239.2
301	164.4	341	188.4	381	213.0	421	239.9
302	165.0	342	189.0	382	213.6	422	240.6
303	165.6	343	189.6	383	214.3	423	241.3
304	166.2	344	190.2	384	214.9	424	242.0
305	166.8	345	190.8	385	215.5	425	242.7
306	167.3	346	191.4	386	216.1	426	243.4
307	167.9	347	192.0	387	216.8	427	244.1
308	168.5	348	192.6	388	217.4	428	244.9
309	169.1	349	193.2	389	218.0	429	245.6
						430	246.3

MALTOSE.

41 *General Gravimetric Method.—Official.*

Proceed as directed under **25** and obtain, from XXX, Table 1, the weight of maltose equivalent to the weight of copper reduced.

Wein Method.—Official.

42 REAGENTS.

The reagents and solutions used are described under **24**.

43 DETERMINATION.

Place 50 cc. of the reagent in a beaker and heat to the boiling point. When boiling briskly, add 25 cc. of the maltose solution containing not more than 0.250 gram of maltose and boil for 4 minutes. Filter immediately through asbestos and determine, by one of the methods given under **26**, **28–33**, respectively, the amount of copper reduced.

Obtain, from **44**, the weight of maltose equivalent to the weight of copper found.

44 TABLE 4.

For the determination of maltose.

[According to Wein[8].]

[Expressed in milligrams.]

COPPER	CUPROUS OXID	MALTOSE	COPPER	CUPROUS OXID	MALTOSE	COPPER	CUPROUS OXID	MALTOSE
31	34.9	26.1	61	68.7	52.2	91	102.4	78.6
32	36.0	27.0	62	69.8	53.1	92	103.6	79.5
33	37.2	27.9	63	70.9	53.9	93	104.7	80.3
34	38.3	28.7	64	72.1	54.8	94	105.8	81.2
35	39.4	29.6	65	73.2	55.7	95	107.0	82.1
36	40.5	30.5	66	74.3	56.6	96	108.1	83.0
37	41.7	31.3	67	75.4	57.4	97	109.2	83.9
38	42.8	32.2	68	76.6	58.3	98	110.3	84.8
39	43.9	33.1	69	77.7	59.2	99	111.5	85.7
40	45.0	33.9	70	78.8	60.1	100	112.6	86.6
41	46.2	34.8	71	79.9	61.0	101	113.7	87.5
42	47.3	35.7	72	81.1	61.8	102	114.8	88.4
43	48.4	36.5	73	82.2	62.7	103	116.0	89.2
44	49.5	37.4	74	83.3	63.6	104	117.1	90.1
45	50.7	38.3	75	84.4	64.5	105	118.2	91.0
46	51.8	39.1	76	85.6	65.4	106	119.3	91.9
47	52.9	40.0	77	86.7	66.2	107	120.5	92.8
48	54.0	40.9	78	87.8	67.1	108	121.6	93.7
49	55.2	41.8	79	88.9	68.0	109	122.7	94.6
50	56.3	42.6	80	90.1	68.9	110	123.8	95.5
51	57.4	43.5	81	91.2	69.7	111	125.0	96.4
52	58.5	44.4	82	92.3	70.6	112	126.1	97.3
53	59.7	45.2	83	93.4	71.5	113	127.2	98.1
54	60.8	46.1	84	94.6	72.4	114	128.3	99.0
55	61.9	47.0	85	95.7	73.2	115	129.6	99.9
56	63.0	47.8	86	96.8	74.1	116	130.6	100.8
57	64.2	48.7	87	97.9	75.0	117	131.7	101.7
58	65.3	49.6	88	99.1	75.9	118	132.8	102.6
59	66.4	50.4	89	100.2	76.8	119	134.0	103.5
60	67.6	51.3	90	101.3	77.7	120	135.1	104.4

TABLE 4.—Continued.

[Expressed in milligrams.]

COPPER	CUPROUS OXID	MALTOSE	COPPER	CUPROUS OXID	MALTOSE	COPPER	CUPROUS OXID	MALTOSE
121	136.2	105.3	166	186.9	145.8	211	237.6	185.9
122	137.4	106.2	167	188.0	146.7	212	238.7	186.8
123	138.5	107.1	168	189.1	147.6	213	239.8	187.7
124	139.6	108.0	169	190.3	148.5	214	240.9	188.6
125	140.7	108.9	170	191.4	149.4	215	242.1	189.5
126	141.9	109.8	171	192.5	150.3	216	243.2	190.4
127	143.0	110.7	172	193.6	151.2	217	244.3	191.2
128	144.1	111.6	173	194.8	152.0	218	245.4	192.1
129	145.2	112.5	174	195.9	152.9	219	246.6	193.0
130	146.4	113.4	175	197.0	153.8	220	247.7	193.9
131	147.5	114.3	176	198.1	154.7	221	248.7	194.8
132	148.6	115.2	177	199.3	155.6	222	249.9	195.7
133	149.7	116.1	178	200.4	156.5	223	251.0	196.6
134	150.9	117.0	179	201.5	157.4	224	252.4	197.5
135	152.0	117.9	180	202.6	158.3	225	253.3	198.4
136	153.1	118.8	181	203.8	159.2	226	254.4	199.3
137	154.2	119.7	182	204.9	160.1	227	255.6	200.2
138	155.4	120.6	183	206.0	160.9	228	256.7	201.1
139	156.5	121.5	184	207.1	161.8	229	257.8	202.0
140	157.6	122.4	185	208.3	162.7	230	258.9	202.9
141	158.7	123.3	186	209.4	163.6	231	260.1	203.8
142	159.9	124.2	187	210.5	164.5	232	261.2	204.7
143	161.0	125.1	188	211.7	165.4	233	262.3	205.6
144	162.1	126.0	189	212.8	166.3	234	263.4	206.5
145	163.2	126.9	190	213.9	167.2	235	264.6	207.4
146	164.4	127.8	191	215.0	168.1	236	265.7	208.3
147	165.5	128.7	192	216.2	169.0	237	266.8	209.1
148	166.6	129.6	193	217.3	169.8	238	268.0	210.0
149	167.7	130.5	194	218.4	170.7	239	269.1	210.9
150	168.9	131.4	195	219.5	171.6	240	270.2	211.8
151	170.0	132.3	196	220.7	172.5	241	271.3	212.7
152	171.1	133.2	197	221.8	173.4	242	272.5	213.6
153	172.3	134.1	198	222.9	174.3	243	273.6	214.5
154	173.4	135.0	199	224.0	175.2	244	274.7	215.4
155	174.5	135.9	200	225.2	176.1	245	275.8	216.3
156	175.6	136.8	201	226.3	177.0	246	277.0	217.2
157	176.8	137.7	202	227.4	177.9	247	278.1	218.1
158	177.9	138.6	203	228.5	178.7	248	279.2	219.0
159	179.0	139.5	204	229.7	179.6	249	280.3	219.9
160	180.1	140.4	205	230.8	180.5	250	281.5	220.8
161	181.3	141.3	206	231.9	181.4	251	282.6	221.7
162	182.4	142.2	207	233.0	182.3	252	283.7	222.6
163	183.5	143.1	208	234.2	183.2	253	284.8	223.5
164	184.6	144.0	209	235.3	184.1	254	286.0	224.4
165	185.8	144.9	210	236.4	185.0	255	287.1	225.3

TABLE 4.—Concluded.

[Expressed in milligrams.]

COPPER	CUPROUS OXID	MALTOSE	COPPER	CUPROUS OXID	MALTOSE	COPPER	CUPROUS OXID	MALTOSE
256	288.2	226.2	271	305.1	239.7	286	322.0	253.1
257	289.3	227.1	272	306.2	240.6	287	323.1	254.0
258	290.5	228.0	273	307.3	241.5	288	324.2	254.9
259	291.6	228.9	274	308.5	242.4	289	325.4	255.8
260	292.7	229.8	275	309.6	243.3	290	326.5	256.6
261	293.8	230.7	276	310.7	244.2	291	327.4	257.5
262	295.0	231.6	277	311.9	245.1	292	328.7	258.4
263	296.1	232.5	278	313.0	246.0	293	329.9	259.3
264	297.2	233.4	279	314.1	246.9	294	331.0	260.2
265	298.3	234.3	280	315.2	247.8	295	332.1	261.1
266	299.5	235.2	281	316.4	248.7	296	333.2	262.0
267	300.6	236.1	282	317.5	249.6	297	334.4	262.8
268	301.7	237.0	283	318.6	250.4	298	335.5	263.7
269	302.8	237.9	284	319.7	251.3	299	336.6	264.6
270	304.0	238.8	285	320.9	252.2	300	337.8	265.5

LACTOSE.

45 *General Gravimetric Method.—Official.*

Proceed as directed under **25** and obtain, from **XXX**, Table 1, the weight of lactose equivalent to the weight of copper reduced.

Soxhlet-Wein Method.—Official.

46 REAGENTS.

The reagents and solutions used are described under **24**.

47 DETERMINATION.

Place 50 cc. of the reagent in a beaker and heat to the boiling point. When boiling briskly, add 100 cc. of the lactose solution containing not more than 0.300 gram of lactose and boil for 6 minutes. Filter immediately through asbestos and determine by one of the methods given under **26**, **28–33**, respectively, the amount of copper reduced. Obtain, from **48**, the weight of lactose equivalent to the weight of copper found.

48

TABLE 5.

For the determination of lactose (Soxhlet-Wein[*]).

[Expressed in milligrams.]

COPPER	LACTOSE	COPPER	LACTOSE	COPPER	LACTOSE	COPPER	LACTOSE	COPPER	LACTOSE
100	71.6	145	105.1	190	139.3	235	173.1	280	208.3
101	72.4	146	105.8	191	140.0	236	173.9	281	209.1
102	73.1	147	106.6	192	140.8	237	174.6	282	209.9
103	73.8	148	107.3	193	141.6	238	175.4	283	210.7
104	74.6	149	108.1	194	142.3	239	176.2	284	211.5
105	75.3	150	108.8	195	143.1	240	176.9	285	212.3
106	76.1	151	109.6	196	143.9	241	177.7	286	213.1
107	76.8	152	110.3	197	144.6	242	178.5	287	213.9
108	77.6	153	111.1	198	145.4	243	179.3	288	214.7
109	78.3	154	111.9	199	146.2	244	180.1	289	215.5
110	79.0	155	112.6	200	146.9	245	180.8	290	216.3
111	79.8	156	113.4	201	147.7	246	181.6	291	217.1
112	80.5	157	114.1	202	148.5	247	182.4	292	217.9
113	81.3	158	114.9	203	149.2	248	183.2	293	218.7
114	82.0	159	115.6	204	150.0	249	184.0	294	219.5
115	82.7	160	116.4	205	150.7	250	184.8	295	220.3
116	83.5	161	117.1	206	151.5	251	185.5	296	221.1
117	84.2	162	117.9	207	152.2	252	186.3	297	221.9
118	85.0	163	118.6	208	153.0	253	187.1	298	222.7
119	85.7	164	119.4	209	153.7	254	187.9	299	223.5
120	86.4	165	120.2	210	154.5	255	188.7	300	224.4
121	87.2	166	120.9	211	155.2	256	189.4	301	225.2
122	87.9	167	121.7	212	156.0	257	190.2	302	225.9
123	88.7	168	122.4	213	156.7	258	191.0	303	226.7
124	89.4	169	123.2	214	157.5	259	191.8	304	227.5
125	90.1	170	123.9	215	158.2	260	192.5	305	228.3
126	90.9	171	124.7	216	159.0	261	193.3	306	229.1
127	91.6	172	125.5	217	159.7	262	194.1	307	229.8
128	92.4	173	126.2	218	160.4	263	194.9	308	230.6
129	93.1	174	127.0	219	161.2	264	195.7	309	231.4
130	93.8	175	127.8	220	161.9	265	196.4	310	232.2
131	94.6	176	128.5	221	162.7	266	197.2	311	232.9
132	95.3	177	129.3	222	163.4	267	198.0	312	233.7
133	96.1	178	130.1	223	164.2	268	198.8	313	234.5
134	96.9	179	130.8	224	164.9	269	199.5	314	235.3
135	97.6	180	131.6	225	165.7	270	200.3	315	236.1
136	98.3	181	132.4	226	166.4	271	201.1	316	236.8
137	99.1	182	133.1	227	167.2	272	201.9	317	237.6
138	99.8	183	133.9	228	167.9	273	202.7	318	238.4
139	100.5	184	134.7	229	168.6	274	203.5	319	239.2
140	101.3	185	135.4	230	169.4	275	204.3	320	240.0
141	102.0	186	136.2	231	170.1	276	205.1	321	240.7
142	102.8	187	137.0	232	170.9	277	205.9	322	241.5
143	103.5	188	137.7	233	171.6	278	206.7	323	242.3
144	104.3	189	138.5	234	172.4	279	207.5	324	243.1

TABLE 5.—Concluded.

COPPER	LACTOSE	COPPER	LACTOSE	COPPER	LACTOSE	COPPER	LACTOSE	COPPER	LACTOSE
325	243.9	340	255.7	355	268.0	370	280.5	385	293.4
326	244.6	341	256.5	356	268.8	371	281.4	386	294.2
327	245.4	342	257.4	357	269.6	372	282.2	387	295.1
328	246.2	343	258.2	358	270.4	373	283.1	388	296.0
329	247.0	344	259.0	359	271.2	374	283.9	389	296.8
330	247.7	345	259.8	360	272.1	375	284.8	390	297.7
331	248.5	346	260.6	361	272.9	376	285.7	391	298.5
332	249.2	347	261.4	362	273.7	377	286.5	392	299.4
333	250.0	348	262.3	363	274.5	378	287.4	393	300.3
334	250.8	349	263.1	364	275.3	379	288.2	394	301.1
335	251.6	350	263.9	365	276.2	380	289.1	395	302.0
336	252.5	351	264.7	366	277.1	381	289.9	396	302.8
337	253.3	352	265.5	367	277.9	382	290.8	397	303.7
338	254.1	353	266.3	368	278.8	383	291.7	398	304.5
339	254.9	354	267.2	369	279.6	384	292.5	399	305.4
								400	306.2

DEXTROSE.

49 *Approximate Volumetric Method for Rapid Work.—Tentative.*

Proceed as directed under **21**. Standardize the reagent against pure dextrose.

50 *Soxhlet Method.—Tentative.*

Proceed as directed under **23**. Under these conditions 100 cc. of the reagent require 0.475 gram of anhydrous dextrose for complete reduction and the formula becomes

$$\frac{100\times0.475}{VW} = \text{per cent of dextrose.}$$

51 *General Gravimetric Method.—Official.*

Proceed as directed under **25** and obtain, from XXX, Table 1, the weight of dextrose equivalent to the weight of copper reduced.

Allihn Gravimetric Method.—Official.

52 REAGENT.

Allihn's Modification of Fehling's Solution.—Prepared by mixing, immediately before use, equal volumes of (**a**) and (**b**).

(**a**) *Copper sulphate solution.*—Dissolve 34.639 grams of copper sulphate ($CuSO_4$. $5H_2O$) in water and dilute to 500 cc.

(**b**) *Alkaline tartrate solution.*—Dissolve 173 grams of Rochelle salts and 125 grams of potassium hydroxid in water and dilute to 500 cc.

53 DETERMINATION.

Place 30 cc. of the copper sulphate solution, 30 cc. of the alkaline tartrate solution, and 60 cc. of water in a beaker and heat to boiling. Add 25 cc. of the solution of the material to be examined, prepared so as not to contain more than 0.25 gram of dextrose, and boil for exactly 2 minutes, keeping the beaker covered. Filter immediately through asbestos, and obtain the weight of copper by one of the methods given under **26**, **28–33**, respectively. The corresponding weight of dextrose is found in **54**.

54

TABLE 6.—ALLIHN'S TABLE[10].

For the determination of dextrose.

[Expressed in milligrams.]

COPPER	CUPROUS OXID	DEXTROSE	COPPER	CUPROUS OXID	DEXTROSE	COPPER	CUPROUS OXID	DEXTROSE
11	12.4	6.6	56	63.0	28.8	101	113.7	51.4
12	13.5	7.1	57	64.2	29.3	102	114.8	51.9
13	14.6	7.6	58	65.3	29.8	103	116.0	52.4
14	15.8	8.1	59	66.4	30.3	104	117.1	52.9
15	16.9	8.6	60	67.6	30.8	105	118.2	53.5
16	18.0	9.0	61	68.7	31.3	106	119.3	54.0
17	19.1	9.5	62	69.8	31.8	107	120.5	54.5
18	20.3	10.0	63	70.9	32.3	108	121.6	55.0
19	21.4	10.5	64	72.1	32.8	109	122.7	55.5
20	22.5	11.0	65	73.2	33.3	110	123.8	56.0
21	23.6	11.5	66	74.3	33.8	111	125.0	56.5
22	24.8	12.0	67	75.4	34.3	112	126.1	57.0
23	25.9	12.5	68	76.6	34.8	113	127.2	57.5
24	27.0	13.0	69	77.7	35.3	114	128.3	58.0
25	28.1	13.5	70	78.8	35.8	115	129.6	58.6
26	29.3	14.0	71	79.9	36.3	116	130.6	59.1
27	30.4	14.5	72	81.1	36.8	117	131.7	59.6
28	31.5	15.0	73	82.2	37.3	118	132.8	60.1
29	32.7	15.5	74	83.3	37.8	119	134.0	60.6
30	33.8	16.0	75	84.4	38.3	120	135.1	61.1
31	34.9	16.5	76	85.6	38.8	121	136.2	61.6
32	36.0	17.0	77	86.7	39.3	122	137.4	62.1
33	37.2	17.5	78	87.8	39.8	123	138.5	62.6
34	38.3	18.0	79	88.9	40.3	124	139.6	63.1
35	39.4	18.5	80	90.1	40.8	125	140.7	63.7
36	40.5	18.9	81	91.2	41.3	126	141.9	64.2
37	41.7	19.4	82	92.3	41.8	127	143.0	64.7
38	42.8	19.9	83	93.4	42.3	128	144.1	65.2
39	43.9	20.4	84	94.6	42.8	129	145.2	65.7
40	45.0	20.9	85	95.7	43.4	130	146.4	66.2
41	46.2	21.4	86	96.8	43.9	131	147.5	66.7
42	47.3	21.9	87	97.9	44.4	132	148.6	67.2
43	48.4	22.4	88	99.1	44.9	133	149.7	67.7
44	49.5	22.9	89	100.2	45.4	134	150.9	68.2
45	50.7	23.4	90	101.3	45.9	135	152.0	68.8
46	51.8	23.9	91	102.4	46.4	136	153.1	69.3
47	52.9	24.4	92	103.6	46.9	137	154.2	69.8
48	54.0	24.9	93	104.7	47.4	138	155.4	70.3
49	55.2	25.4	94	105.8	47.9	139	156.5	70.8
50	56.3	25.9	95	107.0	48.4	140	157.6	71.3
51	57.4	26.4	96	108.1	48.9	141	158.7	71.8
52	58.5	26.9	97	109.2	49.4	142	159.9	72.3
53	59.7	27.4	98	110.3	49.9	143	161.0	72.9
54	60.8	27.9	99	111.5	50.4	144	162.1	73.4
55	61.9	28.4	100	112.6	50.9	145	163.2	73.9

TABLE 6.—ALLIHN'S TABLE.—Continued.

[Expressed in milligrams.]

COPPER	CUPROUS OXID	DEXTROSE	COPPER	CUPROUS OXID	DEXTROSE	COPPER	CUPROUS OXID	DEXTROSE
146	164.4	74.4	191	215.0	97.8	236	265.7	121.7
147	165.5	74.9	192	216.2	98.4	237	266.8	122.3
148	166.6	75.5	193	217.3	98.9	238	268.0	122.8
149	167.7	76.0	194	218.4	99.4	239	269.1	123.4
150	168.9	76.5	195	219.5	100.0	240	270.2	123.9
151	170.0	77.0	196	220.7	100.5	241	271.3	124.4
152	171.1	77.5	197	221.8	101.0	242	272.5	125.0
153	172.3	78.1	198	222.9	101.5	243	273.6	125.5
154	173.4	78.6	199	224.0	102.0	244	274.7	126.0
155	174.5	79.1	200	225.2	102.6	245	275.8	126.6
156	175.6	79.6	201	226.3	103.1	246	277.0	127.1
157	176.8	80.1	202	227.4	103.7	247	278.1	127.6
158	177.9	80.7	203	228.5	104.2	248	279.2	128.1
159	179.0	81.2	204	229.7	104.7	249	280.3	128.7
160	180.1	81.7	205	230.8	105.3	250	281.5	129.2
161	181.3	82.2	206	231.9	105.8	251	282.6	129.7
162	182.4	82.7	207	233.0	106.3	252	283.7	130.3
163	183.5	83.3	208	234.2	106.8	253	284.8	130.8
164	184.6	83.8	209	235.3	107.4	254	286.0	131.4
165	185.8	84.3	210	236.4	107.9	255	287.1	131.9
166	186.9	84.8	211	237.6	108.4	256	288.2	132.4
167	188.0	85.3	212	238.7	109.0	257	289.3	133.0
168	189.1	85.9	213	239.8	109.5	258	290.5	133.5
169	190.3	86.4	214	240.9	110.0	259	291.6	134.1
170	191.4	86.9	215	242.1	110.6	260	292.7	134.6
171	192.5	87.4	216	243.2	111.1	261	293.8	135.1
172	193.6	87.9	217	244.3	111.6	262	295.0	135.7
173	194.8	88.5	218	245.4	112.1	263	296.1	136.2
174	195.9	89.0	219	246.6	112.7	264	297.2	136.8
175	197.0	89.5	220	247.7	113.2	265	298.3	137.3
176	198.1	90.0	221	248.7	113.7	266	299.5	137.8
177	199.3	90.5	222	249.9	114.3	267	300.6	138.4
178	200.4	91.1	223	251.0	114.8	268	301.7	138.9
179	201.5	91.6	224	252.4	115.3	269	302.8	139.5
180	202.6	92.1	225	253.3	115.9	270	304.0	140.0
181	203.8	92.6	226	254.4	116.4	271	305.1	140.6
182	204.9	93.1	227	255.6	116.9	272	306.2	141.1
183	206.0	93.7	228	256.7	117.4	273	307.3	141.7
184	207.1	94.2	229	257.8	118.0	274	308.5	142.2
185	208.3	94.7	230	258.9	118.5	275	309.6	142.8
186	209.4	95.2	231	260.1	119.0	276	310.7	143.3
187	210.5	95.7	232	261.2	119.6	277	311.9	143.9
188	211.7	96.3	233	262.3	120.1	278	313.0	144.4
189	212.8	96.8	234	263.4	120.7	279	314.1	145.0
190	213.9	97.3	235	264.6	121.2	280	315.2	145.5

Table 6.—Allihn's Table.—Continued.

[Expressed in milligrams.]

COPPER	CUPROUS OXID	DEXTROSE	COPPER	CUPROUS OXID	DEXTROSE	COPPER	CUPROUS OXID	DEXTROSE
281	316.4	146.1	326	367.0	170.9	371	417.7	196.3
282	317.5	146.6	327	368.2	171.4	372	418.8	196.8
283	318.6	147.2	328	369.3	172.0	373	420.0	197.4
284	319.7	147.7	329	370.4	172.5	374	421.1	198.0
285	320.9	148.3	330	371.5	173.1	375	422.2	198.6
286	322.0	148.8	331	372.7	173.7	376	423.3	199.1
287	323.1	149.4	332	373.8	174.2	377	424.5	199.7
288	324.2	149.9	333	374.9	174.8	378	425.6	200.3
289	325.4	150.5	334	376.0	175.3	379	426.7	200.8
290	326.5	151.0	335	377.2	175.9	380	427.8	201.4
291	327.4	151.6	336	378.3	176.5	381	429.0	202.0
292	328.7	152.1	337	379.4	177.0	382	430.1	202.5
293	329.9	152.7	338	380.5	177.6	383	431.2	203.1
294	331.0	153.2	339	381.7	178.1	384	432.3	203.7
295	332.1	153.8	340	382.8	178.7	385	433.5	204.3
296	333.3	154.3	341	383.9	179.3	386	434.6	204.8
297	334.4	154.9	342	385.0	179.8	387	435.7	205.4
298	335.5	155.4	343	386.2	180.4	388	436.8	206.0
299	336.6	156.0	344	387.3	180.9	389	438.0	206.5
300	337.8	156.5	345	388.4	181.5	390	439.1	207.1
301	338.9	157.1	346	389.6	182.1	391	440.2	207.7
302	340.0	157.6	347	390.7	182.6	392	441.3	208.3
303	341.1	158.2	348	391.8	183.2	393	442.4	208.8
304	342.3	158.7	349	392.9	183.7	394	443.6	209.4
305	343.4	159.3	350	394.0	184.3	395	444.7	210.0
306	344.5	159.8	351	395.2	184.9	396	445.9	210.6
307	345.6	160.4	352	396.3	185.4	397	447.0	211.2
308	346.8	160.9	353	397.4	186.0	398	448.1	211.7
309	347.9	161.5	354	398.6	186.6	399	449.2	212.3
310	349.0	162.0	355	399.7	187.2	400	450.3	212.9
311	350.1	162.6	356	400.8	187.7	401	451.5	213.5
312	351.3	163.1	357	401.9	188.3	402	452.6	214.1
313	352.4	163.7	358	403.1	188.9	403	453.7	214.6
314	353.5	164.2	359	404.2	189.4	404	454.8	215.2
315	354.6	164.8	360	405.3	190.0	405	456.0	215.8
316	355.8	165.3	361	406.4	190.6	406	457.1	216.4
317	356.9	165.9	362	407.6	191.1	407	458.2	217.0
318	358.0	166.4	363	408.7	191.7	408	459.4	217.5
319	359.1	167.0	364	409.8	192.3	409	460.5	218.1
320	360.3	167.5	365	410.9	192.9	410	461.6	218.7
321	361.4	168.1	366	412.1	193.4	411	462.7	219.3
322	362.5	168.6	367	413.2	194.0	412	463.8	219.9
323	363.7	169.2	368	414.3	194.6	413	465.0	220.4
324	364.8	169.7	369	415.4	195.1	414	466.1	221.0
325	365.9	170.3	370	416.6	195.7	415	467.2	221.6

TABLE 6.—ALLIHN'S TABLE.—Concluded.

[Expressed in milligrams.]

COPPER	CUPROUS OXID	DEXTROSE	COPPER	CUPROUS OXID	DEXTROSE	COPPER	CUPROUS OXID	DEXTROSE
416	468.4	222.2	436	490.9	233.9	456	513.4	245.7
417	469.5	222.8	437	492.0	234.5	457	514.5	246.3
418	470.6	223.3	438	493.1	235.1	458	515.6	246.9
419	471.8	223.9	439	494.3	235.7	459	516.8	247.5
420	472.9	224.5	440	495.4	236.3	460	517.9	248.1
421	474.0	225.1	441	496.5	236.9	461	519.0	248.7
422	475.6	225.7	442	497.6	237.5	462	520.1	249.3
423	476.2	226.3	443	498.8	238.1	463	521.3	249.9
424	477.4	226.9	444	499.9	238.7			
425	478.5	227.5	445	501.0	239.3			
426	479.6	228.0	446	502.1	239.8			
427	480.7	228.6	447	503.2	240.4			
428	481.9	229.2	448	504.4	241.0			
429	483.0	229.8	449	505.5	241.6			
430	484.1	230.4	450	506.6	242.2			
431	485.3	231.0	451	507.8	242.8			
432	486.4	231.6	452	508.9	243.4			
433	487.5	232.2	453	510.0	244.0			
434	488.6	232.8	454	511.1	244.6			
435	489.7	233.4	455	512.3	245.2			

55 REDUCING SUGARS OTHER THAN DEXTROSE.—OFFICIAL.

Proceed as directed under **53** and multiply the weight of dextrose found in **54** by the following factors:

Levulose, 1.093;
Invert sugar, 1.044;
Arabinose, 0.969;
Xylose, 1.017;
Galactose, 1.114.

TOTAL SUGARS[41]—TENTATIVE.

56 PREPARATION OF SOLUTION.

Place 12 grams of the material in a 300 cc. graduated flask, if the substance has an acid reaction, add 1–3 grams of calcium carbonate and boil on a steam bath for 1 hour with 150 cc. of 50 per cent alcohol by volume, using a small funnel in the neck of the flask to condense the vapor. Cool, and allow the mixture to stand several hours, preferably overnight. Make up to volume with neutral 95 per cent alcohol, mix thoroughly, allow to settle, transfer 200 cc. to a beaker with a pipette and evaporate on a steam bath to a volume of 20–30 cc.

Do not evaporate to dryness, a little alcohol in the residue doing no harm. Transfer to a 100 cc. graduated flask, and rinse the beaker thoroughly with water, adding the rinsings to the contents of the flask. Add enough saturated neutral lead acetate solution to produce a flocculent precipitate, shake thoroughly and allow to stand 15 minutes. Make up to the mark with water, mix thoroughly and filter through a dry filter. Add sufficient anhydrous sodium carbonate to the filtrate to precipitate all the lead, again filter through a dry paper and test the filtrate with a little anhydrous sodium carbonate to make sure that all the lead has been removed.

57 REDUCING SUGARS.

Proceed as directed under **25** or **53**, using 25 cc. of the solution (representing 2 grams of the sample), prepared as directed in **56**. Express the results as dextrose or invert sugar.

58 SUCROSE.

Introduce 50 cc. of the solution, prepared as directed in **56**, into a 100 cc. graduated flask, add a piece of litmus paper, neutralize with hydrochloric acid, add 5 cc. of concentrated hydrochloric acid and allow the inversion to proceed at room temperature as directed under **14**. When inversion is complete, transfer the solution to a beaker, neutralize with sodium carbonate, return the solution to the 100 cc. flask, dilute to the mark with water, filter if necessary and determine reducing sugars in 50 cc. of the solution (representing 2 grams of the sample) as directed in **57** and calculate the results as invert sugar. Subtract the per cent of reducing sugars before inversion from the per cent of total sugar after inversion, both calculated as invert sugar, and multiply the difference by 0.95 to obtain the per cent of sucrose present.

Since the insoluble material of grain or cattle food occupies some space in the flask as originally made up, it is necessary to correct for this volume. Results of a large number of determinations on various materials have shown the average volume of 12 grams of material to be 9 cc., and therefore to obtain the true amount of sugars present all results must multiplied by the factor 0.97.

STARCH.

59 *Direct Acid Hydrolysis.—Official.*

(In this method there will be included as starch the pentosans and other carbohydrate bodies present which undergo hydrolysis and conversion into reducing sugars on boiling with hydrochloric acid.)

Stir a quantity of the sample, representing 2.5–3 grams of the dry material, in a beaker with 50 cc. of cold water for an hour. Transfer to a filter and wash with 250 cc. of cold water. Heat the insoluble residue for 2.5 hours with 200 cc. of water and 20 cc. of hydrochloric acid (sp. gr. 1.125) in a flask provided with a reflux condenser. Cool, and nearly neutralize with sodium hydroxid. Complete the volume to 250 cc., filter, and determine the dextrose in an aliquot of the filtrate as directed under **51** or **53**. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

The factor 0.90 is the theoretical ratio between starch and glucose but, according to Noyes[12] and other investigators, the factor 0.93 more nearly approaches the actual yield.

Diastase Method with Subsequent Acid Hydrolysis.—Official.

60 REAGENT.

Malt extract.—Digest 10 grams of fresh, finely ground malt for 2–3 hours at ordinary temperature with 200 cc. of water and filter. Determine the amount of dextrose in a given quantity of the filtrate after boiling with acid, etc., as in the starch determination, and make the proper correction in the subsequent determination.

61 DETERMINATION.

Extract a convenient quantity of the substance (ground to an impalpable powder and representing 4–5 grams of the dry material) on a hardened filter with 5 successive portions of 10 cc. of ether; wash with 150 cc. of 10 per cent alcohol and then with a little strong alcohol. Place the residue in a beaker with 50 cc. of water, immerse the beaker in boiling water, and stir constantly for 15 minutes or until all the starch is gelatinized; cool to 55°C., add 20 cc. of malt extract and maintain at this temperature for an hour. Heat again to boiling for a few minutes, cool to 55°C., add 20 cc. of malt extract and maintain at this temperature for an hour or until the residue treated with iodin shows no blue color upon microscopic examination. Cool, make up directly to 250 cc. and filter. Place 200 cc. of the filtrate in a flask with 20 cc. of hydrochloric acid (sp. gr. 1.125); connect with a reflux condenser and heat in a boiling water bath for 2.5 hours. Cool, nearly neutralize with sodium hydroxid solution, finish the neutralization with sodium carbonate solution and make up to 500 cc. Mix the solution well, pour through a dry filter and determine the dextrose in an aliquot as directed under **51** or **53**. Conduct a blank determination upon the same volume of the malt extract as used upon the sample and correct the weight of reduced copper accordingly. The weight of the dextrose obtained multiplied by 0.90 gives the weight of starch.

PENTOSANS.—OFFICIAL.

62 REAGENT.

Phloroglucin.—Dissolve a small quantity of the phloroglucin in a few drops of acetic anhydrid, heat almost to boiling and add a few drops of concentrated sulphuric acid. A violet color indicates the presence of diresorcin. A phloroglucin which gives more than a faint coloration may be purified by the following method:

Heat in a beaker about 300 cc. of hydrochloric acid (sp. gr. 1.06) and 11 grams of commercial phloroglucin, added in small quantities at a time, stirring constantly until it has almost entirely dissolved. Pour the hot solution into a sufficient quantity of the same hydrochloric acid (cold) to make the volume 1500 cc. Allow it to stand at least overnight, preferably several days, to permit the diresorcin to crystallize. Filter immediately before using. A yellow tint does not interfere with its usefulness. In using it, add the volume containing the required amount to the distillate.

63 DETERMINATION.

Place a quantity of the material, 2–5 grams, chosen so that the weight of phloroglucid obtained shall not exceed 0.300 gram, in a 300 cc. distillation flask, together with 100 cc. of 12 per cent hydrochloric acid (sp. gr. 1.06) and several pieces of recently heated pumice stone. Place the flask on a wire gauze, connect with a condenser, and heat, rather gently at first, and regulate so as to distil over 30 cc. in about 10 minutes, the distillate passing through a small filter paper. Replace the 30 cc. distilled by a like quantity of the dilute acid, added by means of a separatory funnel in such a manner as to wash down the particles adhering to the sides of the flask, and continue the process until the distillate amounts to 360 cc. To the total distillate add gradually a quantity of phloroglucin dissolved in 12 per cent hydrochloric acid and stir thoroughly the resulting mixture. The amount of phloroglucin used should be about double that of the furfural expected. The solution turns first yellow, then green, and very soon an amorphous greenish precipitate appears, which grows darker rapidly, till it becomes finally almost black. Make the solution up to 400 cc. with 12 per cent hydrochloric acid and allow to stand overnight.

Filter the amorphous black precipitate in a tared Gooch crucible having an asbestos mat, wash carefully with 150 cc. of water in such a way that the water is not entirely removed from the crucible until the very last, then dry for 4 hours at the temperature of boiling water, cool and weigh in a weighing bottle, the increase in weight being reckoned as furfural phloroglucid. To calculate the furfural, pentose, or pentosan from the phloroglucid, use the following formulas given by Kröber:

(1) For a weight of phloroglucid, designated by "a" in the following formulas, *under* 0.03 gram,

Furfural = (a + 0.0052) × 0.5170.
Pentoses = (a + 0.0052) × 1.0170.
Pentosans = (a + 0.0052) × 0.8949.

In the above and also in the following formulas, the factor 0.0052 represents the weight of phloroglucid which remains dissolved in the 400 cc. of acid solution.

(2) For a weight of phloroglucid "a" *between* 0.03 *and* 0.300 gram, use Kröber's table, XXX, Table 2, or the following formulas[12]:

Furfural = (a + 0.0052) × 0.5185.
Pentoses = (a + 0.0052) × 1.0075.
Pentosans = (a + 0.0052) × 0.8866.

(3) For a weight of phloroglucid "a" *over* 0.300 gram,

Furfural = (a + 0.0052) × 0.5180.
Pentoses = (a + 0.0052) × 1.0026.
Pentosans = (a + 0.0052) × 0.8824.

64 GALACTAN.—TENTATIVE.

Extract a convenient quantity of the substance, representing 2.5–3 grams of the dry material, on a hardened filter with 5 successive portions of 10 cc. of ether, place the extracted residue in a beaker, about 5.5 cm. in diameter and 7 cm. deep, together with 60 cc. of nitric acid (sp. gr. 1.15), and evaporate the solution to exactly one-third its volume in a water bath at a temperature of 94°–96°C. After standing 24 hours, add 10 cc. of water to the precipitate and allow it to stand another 24 hours. The mucic acid has in the meantime crystallized, but it is mixed with considerable material only partially oxidized by the nitric acid. Filter the solution through filter paper, wash with 30 cc. of water to remove as much of the nitric acid as possible and replace the filter and contents in the beaker. Add 30 cc. of ammonium carbonate solution, consisting of 1 part ammonium carbonate, 19 parts water, and 1 part strong ammonium hydroxid and heat the mixture on a water bath, at 80°C., for 15 minutes, with constant stirring. The ammonium carbonate takes up the mucic acid, forming soluble ammonium mucate. Wash the filter paper and contents several times with hot water by decantation, passing the washings through a filter paper, to which finally transfer the material and thoroughly wash. Evaporate the filtrate to dryness over a water bath, avoiding unnecessary heating which causes decomposition, add 5 cc. of nitric acid (sp. gr. 1.15), stir thoroughly the mixture and allow to stand for 30 minutes. The nitric acid decomposes the ammonium mucate, precipitating the mucic acid; collect this on a tared Gooch or other filter, wash with 10–15 cc. of water, then with 60 cc. of alcohol, and a number of times with ether, dry at the temperature of boiling water for 3 hours and weigh. Multiply the weight of the mucic acid by 1.33, which gives galactose, and multiply this product by 0.9, which gives galactan.

CRUDE FIBER.—OFFICIAL.

65 REAGENTS.

(a) *Dilute sulphuric acid solution.*—Contains exactly 1.25 grams of sulphuric acid (H_2SO_4) in 100 cc. as determined by titration.

(b) *Dilute sodium hydroxid solution.*—Contains exactly 1.25 grams of sodium hydroxid (NaOH) in 100 cc. as determined by titration. This solution should be free, or practically free, from sodium carbonate.

66 DETERMINATION.

Extract a quantity of the substance, representing about 2 grams of the dry material, with ordinary ether, or use the residue from the determination of the ether extract. To this residue in a 500 cc. flask add 200 cc. of the boiling dilute sulphuric acid solution; connect the flask with a reflux condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask, or simply cover a tall conical flask, which is well suited for this determination, with a watch glass or short stemmed funnel, boil at once and continue boiling gently for 30 minutes. A blast of air conducted into the flask will serve to reduce the frothing of the liquid. Filter through linen and wash with boiling water until the washings are no longer acid; rinse the substance back into the flask with 200 cc. of the boiling dilute sodium hydroxid solution, boil at once, and continue boiling gently for 30 minutes as directed above for the treatment with acid, filter at once rapidly and wash with boiling water until the washings are neutral. The last filtration may be performed upon a Gooch crucible, a linen filter, or a tared filter paper. If a linen filter is used, rinse the crude fiber, after washing is completed, into a flat-bottomed platinum dish by means of a jet of water; evaporate to dryness on a steam bath, dry to constant weight at 110°C., weigh, incinerate completely and weigh again. The loss in weight is considered to be crude fiber. If a tared filter paper is used, weigh in a weighing bottle. In any case the crude fiber after drying to constant weight at 110°C. must be incinerated and the amount of the ash deducted from the original weight.

67 WATER-SOLUBLE ACIDITY OF FEEDS.—TENTATIVE.

Weigh 10 grams of the sample into a shaking bottle, add 200 cc. of water, and shake for 15 minutes. Filter the extract through a folded filter and take a 20 cc. aliquot (equivalent to 1 gram of sample) for the titration. Dilute with 50 cc. of water and titrate with N/10 sodium hydroxid, using phenolphthalein as indicator.

In reporting the acidity of feeds, state the results in terms of cc. of N/10 sodium hydroxid required for neutralization.

DETERMINATION OF HYDROCYANIC ACID FORMED BY THE HYDROLYSIS OF GLUCOSIDES IN BEANS[14].

68 *Acid Titration Method.—Tentative.*

Grind the sample to pass a 20 mesh sieve. Introduce 10–20 grams of the ground sample into an 800 cc. Kjeldahl flask, add 100 cc. of water and macerate at room temperature for 2 hours. Add 100 cc. of water and distil with steam, collecting the distillate in 20 cc. of N/50 silver nitrate solution acidified with 1 cc. of concentrated nitric acid. During the distillation, adjust the apparatus so that the tip of the condenser dips below the surface of the liquid in the receiver. When 150 cc. of distillate have passed over, filter the contents of the receiver through a Gooch, wash the receiver and Gooch with a little water and titrate the excess of silver nitrate in the combined filtrate and washings with N/50 potassium thiocyanate solution, using ferric alum as indicator. One cc. of N/50 silver nitrate solution is equivalent to 0.54 mg. of hydrocyanic acid (HCN).

69 *Alkaline Titration Method.—Tentative.*

Macerate and distil 10–20 grams of the sample as described in 68 except that the distillate is collected in a vessel containing 0.5 gram of sodium hydroxid dissolved in 20 cc. of water, the tip of the condenser dipping below the surface of the liquid in the receiver during the distillation. When 150 cc. of distillate have passed over, titrate the liquid in the receiver with N/50 silver nitrate solution, adding the standard solution slowly, drop by drop, stirring constantly, until the first permanent turbidity appears. One cc. of N/50 silver nitrate solution is equivalent to 1.08 mg. of hydrocyanic acid (HCN).

70 *Prussian Blue Method.—Tentative.*

Macerate and distil 10–20 grams of the sample as described in 69, using the sodium hydroxid solution in the receiver, and dilute the distillate to 200 cc. in a graduated flask. Concentrate 20 cc. of this solution, which must contain a slight excess of free sodium hydroxid, in a 200 cc. round-bottomed flask attached to a vacuum pump and condenser, heating the flask in a water bath below 70°C. An adapter may be used to avoid loss by spattering. When the volume has been reduced to 1 cc. or less, add 0.2–0.5 cc. of freshly prepared 3 per cent ferrous sulphate solution and about 0.5 gram of potassium fluorid. Exhaust the flask at once by means of a vacuum pump. Mix the contents by rotating the flask. After 5–10 minutes detach the flask and acidify the mixture with 30 per cent nitric acid. The blue color usually appears at once, although in case traces only are present it is sometimes necessary to warm to about 50°C. in a water bath. Dilute the resulting suspension of Prussian blue to a convenient volume, and compare the color with a standard Prussian blue mixture, prepared as above from a standard solution containing 1 mg. of potassium cyanid diluted to 25 cc., preferably using a Duboscq colorimeter for the comparison.

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VIII. SACCHARINE PRODUCTS.

1 PREPARATION OF SAMPLE.—OFFICIAL.

(a) *Liquids* (*molasses, sirups, etc.*).—Mix materials of this class thoroughly. If crystals of sugar are present, dissolve them either by heating gently or by weighing the whole mass, then adding water, heating until completely dissolved and after cooling, reweighing. Calculate all results to the weight of the original substance.

(b) *Semi-solids* (*jellies, jams, etc.*).—Weigh 50 grams of the sample into a 250 cc. graduated flask. Treat with water, fill to the mark and mix thoroughly. If insoluble material remains, mix uniformly by shaking before taking aliquots for the various determinations.

(c) *Solids* (*sugar, confectionery, etc.*).—Grind and mix thoroughly materials of this class to secure uniform samples.

MOISTURE.

DRYING METHODS.

2 SUGARS.—OFFICIAL.

Dry 2–5 grams in a flat dish (nickel, platinum, or aluminium) at the temperature of boiling water for 10 hours; cool in a desiccator and weigh; then dry again for an hour or until there is only a slight change in weight.

With some sugars, more especially those of large grain, there is danger of occlusion and retention of water. The International Commission for Unifying Methods of Sugar Analysis prescribes drying at 105°–110°C. for normal beet sugars. This temperature is sufficient to expel the last traces of occluded water and is not attended with sufficient decomposition to affect the weight of the product. The drying temperature should never exceed 110°C[1].

MASSECUITES, MOLASSES, AND OTHER LIQUID AND SEMILIQUID PRODUCTS.

3 *Drying upon Pumice Stone.—Official.*

Prepare pumice stone of two grades of fineness, one of which will pass through a 1 mm. sieve, the other through a 6 mm. sieve but not a 1 mm. sieve. Make the determination in flat metallic dishes or in shallow, flat-bottomed weighing bottles. Place a layer of the fine pumice stone, 3 mm. in thickness, on the bottom of the dish, then a layer of the coarse pumice stone 6–10 mm. in thickness, dry and weigh. Dilute the sample with a weighed portion of water so that the diluted material shall contain 20–30 per cent of solid matter. Weigh into the dish, prepared as described above, an amount of the diluted sample to yield, approximately, 1 gram of dry matter. If this weighing can not be made rapidly, use a weighing bottle provided with a cork through which a pipette passes. Dry in vacuo at 70°C. to constant weight, making trial weighings at intervals of 2 hours. For substances containing little or no levulose or other readily decomposable substance, the drying may be made in a water oven at the temperature of boiling water.

4 *Drying upon Quartz Sand.—Official.*

Digest pure quartz sand with strong hydrochloric acid, wash, dry and ignite. Preserve in a stoppered bottle.

Place 6–7 grams of the prepared sand and a short stirring rod in a flat-bottomed dish. Dry thoroughly, cool in a desiccator and weigh. Then add 3–4 grams of the molasses, mix with the sand (if necessary to thoroughly incorporate the two, add a little water), dry in a water oven at the temperature of boiling water for 8–10 hours, stirring at intervals of an hour, cool in a desiccator and weigh. Stir, heat again for an hour, cool and weigh. Repeat the heating and weighing until the loss of water in an hour is not greater than 3 mg.

AREOMETRIC METHODS.

(Not applicable to low-grade sugar products, molasses and other materials containing large amounts of non-sugar solids.)

SPECIFIC GRAVITY, WATER AND TOTAL SOLIDS.

5 *By Means of a Spindle.—Official.*

The density of juices, sirups, etc., is most conveniently determined by means of the Brix hydrometer. For rough work, or where less accuracy is desired, the Baumé hydrometer may be used. The Brix spindle should be graduated to tenths. The range of each individual spindle should be as limited as possible. The solution should be as nearly as practicable of the same temperature as the air at the time of reading, and, if the variation from the temperature of the graduation of the spindle amounts to more than 1°, a correction must be applied according to the table under **6**. A similar table of corrections based upon Brix saccharometers, standard at 20°C., is given in **XXX**, Table 9. Before taking the density of a juice, allow it to stand in the cylinder until all air bubbles have escaped, and until all fatty or waxy matter has come to the surface and been skimmed off. The cylinder should be large enough in diameter to allow the hydrometer to come to rest without touching the sides. A table of specific gravities at $\frac{20°C.}{4°}$ and of per cents by weight of sucrose is given in **XXX**, Table 3. A table for the comparison of specific gravities at $\frac{17.5°C.}{17.5°}$, degrees Brix (per cent by weight of sucrose), and degrees Baumé (modulus 146.78), is given under **8**.

A similar table for the comparison of specific gravities at $\frac{20°C.}{20°}$ and $\frac{20°C.}{4°}$, degrees Brix, and degrees Baumé (modulus 145), is given in **XXX**, Table 10.

If the sample is too dense to determine the density directly, dilute a weighed portion with a weighed quantity of water, or dissolve a weighed portion and dilute to a known volume with water.

In the first instance the per cent of total solids is calculated by the following formula:

Per cent of solids in the undiluted material $= \frac{WS}{w}$ in which

S = per cent of solids in the diluted material;
W = weight of the diluted material;
w = weight of the sample taken for dilution.

When the dilution is made to a definite volume, the following formula is to be used:

Per cent of solids in the undiluted material $= \frac{VDS}{W}$ in which

V = volume of the diluted solution at a given temperature;
D = specific gravity of the diluted solution at the same temperature;
S = per cent of solids in the diluted solution at the same temperature;
W = weight of the sample taken for dilution at the same temperature.

If the spindle reading be made at any other temperature than 17.5°C., the result should be corrected as directed under **6**. For spindles standard at 20°C., corrections should be made according to **XXX**, Table 9.

6 TABLE 7.

For correction of the readings of the Brix spindle when made at other than the standard temperature, 17.5°C.

(For temperatures below 17.5°C. the correction is to be subtracted.)

TEMPERATURE	DEGREE BRIX OF THE SOLUTION												
	0	5	10	15	20	25	30	35	40	50	60	70	75
°C.													
0	0.17	0.30	0.41	0.52	0.62	0.72	0.82	0.92	0.98	1.11	1.22	1.25	1.29
5	0.23	0.30	0.37	0.44	0.52	0.59	0.65	0.72	0.75	0.80	0.88	0.91	0.94
10	0.20	0.26	0.29	0.33	0.36	0.39	0.42	0.45	0.48	0.50	0.54	0.58	0.61
11	0.18	0.23	0.26	0.28	0.31	0.34	0.36	0.39	0.41	0.43	0.47	0.50	0.53
12	0.16	0.20	0.22	0.24	0.26	0.29	0.31	0.33	0.34	0.36	0.40	0.42	0.46
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39
14	0.12	0.15	0.16	0.17	0.18	0.19	0.21	0.22	0.22	0.23	0.26	0.28	0.32
15	0.09	0.11	0.12	0.14	0.14	0.15	0.16	0.17	0.16	0.17	0.19	0.21	0.25
16	0.06	0.07	0.08	0.09	0.10	0.10	0.11	0.12	0.12	0.12	0.14	0.16	0.18
17	0.02	0.02	0.03	0.03	0.03	0.04	0.04	0.04	0.04	0.04	0.05	0.05	0.06
18	0.02	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.02
19	0.06	0.08	0.08	0.09	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.08	0.06
20	0.11	0.14	0.15	0.17	0.17	0.18	0.18	0.18	0.19	0.19	0.18	0.15	0.11
21	0.16	0.20	0.22	0.24	0.24	0.25	0.25	0.25	0.26	0.26	0.25	0.22	0.18
22	0.21	0.26	0.29	0.31	0.31	0.32	0.32	0.32	0.33	0.34	0.32	0.29	0.25
23	0.27	0.32	0.35	0.37	0.38	0.39	0.39	0.39	0.40	0.42	0.39	0.36	0.33
24	0.32	0.38	0.41	0.43	0.44	0.46	0.46	0.47	0.47	0.50	0.46	0.43	0.40
25	0.37	0.44	0.47	0.49	0.51	0.53	0.54	0.55	0.55	0.58	0.54	0.51	0.48
26	0.43	0.50	0.54	0.56	0.58	0.60	0.61	0.62	0.62	0.66	0.62	0.58	0.55
27	0.49	0.57	0.61	0.63	0.65	0.68	0.68	0.69	0.70	0.74	0.70	0.65	0.62
28	0.56	0.64	0.68	0.70	0.72	0.76	0.76	0.78	0.78	0.82	0.78	0.72	0.70
29	0.63	0.71	0.75	0.78	0.79	0.84	0.84	0.86	0.86	0.90	0.86	0.80	0.78
30	0.70	0.78	0.82	0.87	0.87	0.92	0.92	0.94	0.94	0.98	0.94	0.88	0.86
35	1.10	1.17	1.22	1.24	1.30	1.32	1.33	1.35	1.36	1.39	1.34	1.27	1.25
40	1.50	1.61	1.67	1.71	1.73	1.79	1.79	1.80	1.82	1.83	1.78	1.69	1.65
50	...	2.65	2.71	2.74	2.78	2.80	2.80	2.80	2.80	2.79	2.70	2.56	2.51
60	...	3.87	3.88	3.88	3.88	3.88	3.88	3.88	3.90	3.82	3.70	3.43	3.41
70	...	5.17	5.18	5.20	5.14	5.13	5.10	5.08	5.06	4.90	4.72	4.47	4.35
80	...	...	6.62	6.59	6.54	6.46	6.38	6.30	6.26	6.06	5.82	5.50	5.33
90	...	...	8.26	8.16	8.06	7.97	7.83	7.71	7.58	7.30	6.96	6.58	6.37
100	...	...	10.01	9.87	9.72	9.56	9.39	9.21	9.03	8.64	8.22	7.76	7.42

Example.—A sugar solution shows a reading of 30.2° Brix at 30°C. To find the necessary correction for the conversion of this reading to the reading which would have been obtained if the observation had been made at 17.5°C., find the vertical column in the table headed 30° Brix, which is the nearest to the observed reading. Follow down this column until the number is reached which is opposite to the temperature of observation—in this case 30°. The number found, 0.92, is to be added to the observed reading.

7 *By Means of a Pycnometer.—Official.*

(a) *By specific gravity at* $\frac{20^\circ\text{C.}}{4^\circ}$—Determine the specific gravity of the solution at $\frac{20^\circ\text{C}}{4^\circ}$ by means of a pycnometer and ascertain the corresponding per cent by weight of sucrose from XXX, Table 3. When the density of the substance is too high for a direct determination, dilute and calculate the sucrose content of the original material as directed under **5**.

(b) *By specific gravity at* $\frac{17.5°C}{17.5°}$ —**Proceed as directed under (a), the determinations of specific gravity being made at $\frac{17.5°C.}{17.5°}$ instead of at $\frac{20°C.}{4°}$ Ascertain the corresponding per cent by weight of sucrose from 8.**

8

Table 8.

For the comparison of specific gravities at $\frac{17.5°C.}{17.5°}$, degrees Brix and degrees Baumé.

$$\text{Degree Baumé} = 146.78 - \frac{146.78}{\text{sp. gr.}}$$

DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ	DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ	DEGREE BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY	DEGREE BAUMÉ
1.0	1.00388	0.6	33.0	1.14423	18.5	65.0	1.31989	35.6
2.0	1.00779	1.1	34.0	1.14915	19.05	66.0	1.32601	36 1
3.0	1.01173	1.7	35.0	1.15411	19.6	67.0	1.33217	36.6
4.0	1.01570	2.3	36.0	1.15911	20.1	68.0	1.33836	37.1
5.0	1.01970	2.8	37.0	1.16413	20.7	69.0	1.34460	37.6
6.0	1.02373	3.4	38.0	1.16920	21.2	70.0	1.35088	38.1
7.0	1.02779	4.0	39 0	1.17430	21.8	71.0	1.35720	38.6
8.0	1.03187	4.5	40.0	1.17943	22.3	72.0	1.36355	39.1
9.0	1.03599	5.1	41.0	1.18460	22.9	73.0	1.36995	39.6
10.0	1.04014	5.7	42.0	1.18981	23.4	74.0	1.37639	40.1
11.0	1.04431	6.2	43.0	1.19505	23.95	75.0	1.38287	40.6
12.0	1.04852	6.8	44.0	1.20033	24.5	76.0	1.38939	41.1
13.0	1.05276	7.4	45.0	1.20565	25.0	77.0	1.39595	41.6
14.0	1.05703	7.9	46.0	1.21100	25.6	78.0	1.40254	42.1
15.0	1.06133	8.5	47.0	1.21639	26.1	79.0	1.40918	42.6
16.0	1.06566	9.0	48.0	1.22182	26.6	80.0	1.41586	43.1
17.0	1.07002	9.6	49.0	1.22728	27.2	81.0	1.42258	43.6
18.0	1.07441	10.1	50.0	1.23278	27.7	82.0	1.42934	44.1
19.0	1.07884	10.7	51.0	1.23832	28.2	83.0	1.43614	44.6
20.0	1.08329	11.3	52.0	1.24390	28.8	84.0	1.44298	45.1
21.0	1.08778	11.8	53.0	1.24951	29.3	85.0	1.44986	45.5
22.0	1.09231	12.4	54.0	1.25517	29.8	86.0	1.45678	46.0
23.0	1.09686	13.0	55.0	1.26086	30.4	87.0	1.46374	46.5
24.0	1.10145	13.5	56.0	1.26658	30.9	88.0	1.47074	47.0
25.0	1.10607	14.1	57.0	1.27235	31.4	89.0	1.47778	47.45
26.0	1.11072	14.6	58.0	1.27816	31.9	90.0	1.48486	47.9
27.0	1.11541	15.2	59.0	1.28400	32.5	91.0	1.49199	48.5
28.0	1.12013	15.7	60.0	1.28989	33.0	92.0	1.49915	48.9
29.0	1.12488	16.3	61.0	1.29581	33.5	93.0	1.50635	49.4
30.0	1.12967	16.8	62.0	1.30177	34.0	94.0	1.51359	49.8
31.0	1.13449	17.4	63.0	1.30777	34.5	95.0	1.52087	50.3
32.0	1.13934	17.95	64.0	1.31381	35.1			

When the number expressing the specific gravity found by analysis falls between the numbers given in the above table, the exact equivalent in degrees Brix or Baumé is found by a simple calculation.

Example.—The pycnometer shows the specific gravity of a certain sirup to be 1.20909. The table shows that the corresponding degree Brix is between 45.0 and 46.0. Subtracting the specific gravity of a solution of 45° Brix from the corresponding figure for 46°, we have (expressing the specific gravities as whole numbers) 121,100 − 120,565 = 535, the difference in specific gravity for 1° Brix at this point in the table. Subtracting the specific gravity corresponding to 45° from the specific gravity found by analysis, we have 120,909 − 120,565 = 344; $\frac{344}{535}$ = 0.64, the fraction of 1° Brix more than 45°. The degree Brix, corresponding to a sp. gr. of 1.20909, is therefore 45.64.

9 *REFRACTOMETER METHOD.—OFFICIAL.*

(Applicable only to liquid samples containing no undissolved solids.)

Determine the refractive index of the solution at 28°C. and obtain the corresponding percentage of dry substance from **XXX**, Table 5. If the refractive index is obtained at a temperature other than 28°C., correct the result as indicated in **XXX**, Table 6. If the solution is too dark to be read in the instrument, dilute with a concentrated sugar solution. Water should never be used for this purpose. Mix weighed amounts of the solution under examination and a solution of pure sugar of about the same strength, and obtain the amount of dry substance in the former by the following formula:

$$x = \frac{(A + B)\,C - BD}{A} \text{ in which}$$

x = per cent of dry substance to be found;

A = weight in grams of the material mixed with B;

B = weight in grams of pure sugar solution employed in the dilution;

C = per cent of dry substance in the mixture of A and B obtained from the refractive index;

D = per cent of dry substance in the pure sugar solution obtained from its refractive index.

ASH.

10 *Method I.—Official.*

Heat 5–10 grams of the sample in a 50–100 cc. platinum dish at 100°C. until the water is expelled, add a few drops of pure olive oil and heat slowly over a flame until swelling ceases. Then place the dish in a muffle and heat at low redness until a white ash is obtained.

11 *Method II.—Official*

Carbonize the mass at a low heat, dissolve the soluble salts in hot water, burn the residual mass as directed in **10**, add the solution of soluble salts, and evaporate to dryness at 100°C., ignite gently, cool in a desiccator and weigh.

12 *Method III.—Official.*

Saturate the sample with sulphuric acid, dry, ignite gently, then burn in a muffle at low redness. Deduct one-tenth of the weight of the ash and calculate the per cent.

13 QUANTITATIVE ANALYSIS OF THE ASH.—OFFICIAL.

Proceed as directed under **11**.

14 SOLUBLE AND INSOLUBLE ASH.—OFFICIAL.

Ash the material as directed under **10** or **11**. Add water to the ash in the platinum dish, heat nearly to boiling, filter through an ashless filter and wash with hot water until the combined filtrate and washings measure about 60 cc. Return the filter and contents to the platinum dish, ignite carefully, cool and weigh. Calculate the percentages of water-soluble and water-insoluble ash.

15 ALKALINITY OF THE SOLUBLE ASH.—OFFICIAL.

Cool the filtrate from **14** and titrate with N/10 hydrochloric acid, using methyl orange as an indicator.

Express the alkalinity in terms of the number of cc. of N/10 acid per 1 gram of the sample.

16 ALKALINITY OF THE INSOLUBLE ASH.—OFFICIAL.

Add an excess of N/10 hydrochloric acid (usually 10–15 cc.) to the ignited insoluble ash in the platinum dish, under **14**, heat to boiling over an asbestos plate, cool and titrate the excess of hydrochloric acid with N/10 sodium hydroxid, using methyl orange as an indicator.

Express the alkalinity in terms of the number of cc. of N/10 acid per 1 gram of the sample.

17 MINERAL ADULTERANTS IN THE ASH.—TENTATIVE.

In a large porcelain evaporating dish, mix 100 grams of molasses, sirup, honey, or the confectionery solution prepared as directed under **1 (b)** with about 35 grams of concentrated sulphuric acid and evaporate to a sirupy consistency. Pass an electric current through it while stirring by placing one platinum electrode in the bottom of the dish near one side and attaching the other to the lower end of the glass rod with which the contents are stirred. Begin with a current of about 1 ampere and gradually increase to 4 (modified from method of Budde and Schou[2] for determining nitrogen electrolytically). In 10–15 minutes the mass is reduced to a fine dry char, which may be readily burned to a white ash in the original dish over a free flame or in a muffle.

This method[3] is preferred to the ordinary method of heating with sulphuric acid, especially in the case of molasses, because, if properly manipulated, the material comes quietly into the form of a very finely divided char or powder, especially adapted for subsequent quick ignition.

If an electric current is not available, treat in a large porcelain dish 100 grams of the saccharine solution, evaporated to a sirupy consistency, with sufficient concentrated sulphuric acid to thoroughly carbonize the mass, and ignite in the usual manner.

The following adulterants may be present: salts of tin, used in molasses to bleach; mineral pigments, such as chromate of lead in yellow confectionery; oxid of iron, sometimes used to simulate the color of chocolate; and copper. These elements may be detected by the usual qualitative tests.

18 NITROGEN.—OFFICIAL.

Determine nitrogen in 5 grams of the material as directed under **I, 18, 21** or **23**, using a larger quantity of the sulphuric acid if necessary for complete digestion.

SUCROSE.

19 *Method I.—Official.*

(Substances in which the volume of the combined insoluble matter and precipitate from clarifying agents is less than 1 cc. from 26 grams.)

Determine sucrose by polarization before and after inversion, as directed under **VII, 14**.

All products which contain dextrose or other reducing sugars in the crystalline form, or in supersaturated solution, exhibit the phenomenon of birotation. The constant rotation only should be employed in the Clerget formula, and to obtain this the solutions prepared for direct polarization should be allowed to stand overnight before making the reading. If it is desired to make the direct reading immediately, the birotation may be destroyed by heating the neutral solution to boiling for a few minutes or by adding a few drops of strong ammonium hydroxid before completing the volume.

20 *Method II.* (*Double dilution method*[4].)—*Official.*

(Substances in which the volume of the combined insoluble matter and precipitate from clarifying agents is more than 1 cc. from 26 grams.)

Weigh out a half normal weight of the sample and make up the solution to 100 cc., employing the appropriate clarifier (basic lead acetate for dark colored confectionery or molasses and alumina cream for light colored confectionery). Also weigh out a normal weight of the sample and make up a second solution with the clarifier to 100 cc. Filter and obtain direct polariscopic readings of both solutions. Invert each solution as directed under **VII, 14**, and obtain its invert reading.

The true direct polarization of the sample is the product of the two direct readings divided by their difference.

The true invert polarization is the product of the two invert readings divided by their difference.

Calculate the sucrose from the true polarizations thus obtained by one of the formulas given under **VII, 14**.

COMMERCIAL GLUCOSE (APPROXIMATE).

21 *Method I.—Official.*

(Substances containing little or no invert sugar.)

Commercial glucose can not be determined accurately owing to the varying amounts of dextrin, maltose, and dextrose present in this product. However, in sirups, in which the amount of invert sugar is so small as not to appreciably affect the result, commercial glucose may be estimated approximately by the following formula[7]:

$$G = \frac{(a - S)\,100}{175} \text{ in which}$$

G = per cent of commercial glucose;
a = direct polarization;
S = per cent of cane sugar.

Express the results in terms of commercial glucose polarizing +175°V.

22 *Method II.—Official.*

(Substances containing invert sugar[5].)

Prepare an inverted half normal solution of the substance as directed under **VII, 14**, except that after inversion the solution is cooled, made neutral to phenolphthalein with sodium hydroxid solution, slightly acidified with hydrochloric acid, and treated with 5–10 cc. of alumina cream before making up to the mark. Filter and polarize at 87°C. in a 200 mm. jacketed tube. Multiply the reading by 200 and divide by the factor 163 to express the amount of glucose present in terms of glucose polarizing +175°V.

23 REDUCING SUGARS.

Determine as either dextrose or invert sugar as directed under **VII, 49, 50, 51, 53**, or **21, 23, 25, 35** or **38**.

24 STARCH.—TENTATIVE.

Measure 25 cc. of a solution or uniform mixture, prepared as directed in **1 (b)**, (representing 5 grams of the sample), into a 300 cc. beaker, or introduce 5 grams of the finely ground sample (previously extracted with ether if the sample contains much fat) into the beaker, add sufficient water to make the volume 100 cc., heat to about 60°C. (avoiding if possible gelatinizing the starch) and allow to stand for about an hour, stirring frequently to secure complete solution of the sugars. Transfer to a stout wide-mouthed bottle, rinse the beaker with a little warm water, cool, add an equal volume of 95 per cent alcohol, mix and allow to stand at least an hour. Centrifugalize until the precipitate is closely packed on the bottom of the bottle and decant the supernatant liquid through a hardened filter. Wash the precipitate with successive 50 cc. portions of 50 per cent alcohol by centrifugalizing and decanting through the filter until 3 or 4 drops of the washings give no test for sugar with alphanaphthol as described under **66**. Transfer the residue from the bottle and the hardened filter to a large flask and determine starch as directed under **VII, 59**.

ETHER EXTRACT IN CONFECTIONERY.

25 *Continuous Extraction.—Tentative.*

(1) Measure 25 cc. of a 20 per cent mixture or solution, prepared as directed under **1 (b)**, into a very thin, readily frangible, glass evaporating shell (*Hofmeister Schälchen*), containing 5–7 grams of freshly ignited asbestos fiber; or (2) If possible to obtain a uniform sample, weigh 5 grams of the mixed finely divided sample into a dish, and wash with water upon the asbestos in the evaporating shell, using, if necessary, a small portion of the asbestos fiber on a stirring rod to transfer the last traces of the sample from the dish to the shell. Dry to constant weight at 100°C., cool, wrap loosely in smooth paper, crush into rather small fragments between the fingers, transfer carefully the crushed mass, including the paper, to an extraction tube or a fat extraction cartridge. A thin lead disk (bottle cap) may be substituted for the glass shell. The disk may then be cut into small pieces and placed in the extraction tube. Extract with anhydrous ether or petroleum ether (b. p. 45°–60°C. and without weighable residue) in a continuous extraction apparatus for at least 25 hours. In most cases it is advisable to remove the substance from the extractor after the first 12 hours, grind with sand to a fine powder and re-extract for the remaining 13 hours. Transfer the extract to a tared flask, evaporate the solvent and dry to constant weight in an oven at 100°C.

26 *Roese-Gottlieb Method.—Tentative.*

Substances such as butter-scotch invariably yield extremely inaccurate results by the above method. In such cases introduce 4 grams of the material, or an amount of a uniform solution equivalent to this amount of the dry substance, into a Röhrig tube or similar apparatus, make up to a volume of 10 cc. with water, add 1.25 cc. of concentrated ammonium hydroxid and mix thoroughly. Add 10 cc. of 95 per cent alcohol and mix. Then add 25 cc. of washed ether and shake vigorously for half a minute; then add 25 cc. of petroleum ether (b. p. below 60°C.) and shake again for half a minute. Allow to stand for 20 minutes or until the separation of the liquids is complete. Draw off as much as possible of the ether-fat solution (usually 0.5–0.8 cc. will be left) into a weighed flask through a small, rapid filter. The flask should be weighed with a similar one as a counterpoise. Again extract the liquid remaining in the tube, this time with 15 cc. each of ether and petroleum ether, shake vigorously half a minute with each, and allow to settle. Proceed as above, washing the tip of the spigot and the filter with a few cc. of a mixture of equal parts of the 2 ethers (previously mixed and free from deposited water). For absolutely exact results the extraction must be repeated. This third extraction usually yields not more than about 1 mg. of fat, if the previous ether-fat solutions have been drawn off closely, or an amount averaging about 0.02 per cent on a 4 gram charge. Evaporate the ether slowly on a steam bath, then dry the fat in a boiling water oven until the loss in weight ceases. Test the purity of the fat by dissolving in a little petroleum ether. Should a residue remain, wash the fat out completely with petroleum ether, dry the residue, weigh and deduct the weight.

27 PARAFFIN IN CONFECTIONERY.—TENTATIVE.

Add to the ether extract in the flask, as above obtained, 10 cc. of 95 per cent alcohol and 2 cc. of sodium hydroxid solution (1 to 1), connect the flask with a reflux condenser, and heat for an hour on a water bath, or until saponification is complete. Remove the condenser and allow the flask to remain on the bath until the alcohol is evaporated and the residue is dry. Dissolve the residue as completely as possible in about 40 cc. of water and heat on the bath, shaking frequently. Wash into a separatory funnel, cool and extract with 4 successive portions of petroleum ether, which are collected in a tared flask or capsule. Evaporate the petroleum ether and dry in the oven to constant weight.

Any phytosterol or cholesterol present in the fat would be extracted with the paraffin. The amount is so insignificant that it may be disregarded generally. The character of the final residue should, however, be confirmed by determining its melting point, specific gravity and refractive index.

28 ALCOHOL IN SIRUPS USED IN CONFECTIONERY ("BRANDY DROPS").—OFFICIAL.

Collect in a beaker the sirup from a sufficient number of pieces to yield 30–50 grams of sirup. Strain the sirup into a tared beaker and weigh. Introduce the sirup into a 250–300 cc. distillation flask, dilute with half its volume of water, attach the flask to a vertical condenser and distil almost 50 cc., or as much of the liquid as possible without causing charring. Foaming may be prevented by adding to the contents of the distillation flask a little tannin, or a piece of paraffin about the size of a pea. Cool the distillate, make up to volume with water, mix well and ascertain the specific gravity of the liquid by means of a pycnometer. Obtain the corresponding weight of alcohol in the 50 cc. of distillate from XXX, Table 7. Calculate the per cent by weight of alcohol in the candy filling.

29 COLORING MATTER.—TENTATIVE.

Proceed as directed under X.

30 METALS.—TENTATIVE.

Proceed as directed under XI.

HONEY[4].

31 PREPARATION OF SAMPLE.—OFFICIAL.

(a) *Liquid or strained honey.*—If the sample is free from granulation, mix thoroughly by stirring or shaking before drawing weighed portions for the analytical determination. If the honey is granulated, place the container, having the stopper loose, in a water bath and heat at a temperature not exceeding 50°C. until the sugar crystals dissolve; mix thoroughly, cool and weigh portions for the analytical determinations. If sediment such as wax, sticks, bees, particles of comb, etc., is present, heat the sample to 40°C. in a water bath and filter through cheese-cloth before weighing portions for analysis.

(b) *Comb honey.*—Cut across the top of the comb, if sealed, and separate completely from the comb by straining through a 40 mesh sieve. When portions of the comb or wax pass through the sieve, heat the sample as in (a) and strain through cloth. If the honey is granulated in the comb, heat until the wax is liquefied, stir, cool, remove the wax and take the clear liquid for analysis.

32 MOISTURE.—OFFICIAL.

Weigh 2 grams of the sample into a tared, flat-bottomed aluminium dish, having a diameter of about 60 mm. and containing 10–15 grams of fine quartz sand, which has been previously washed, dried and ignited, and a small glass stirring rod; add 5–10 cc. of water and thoroughly incorporate with the sand and honey mixture by means of the rod; dry the dish and its contents to constant weight in a vacuum oven at a temperature not exceeding 70°C.

33 ASH.—OFFICIAL.

Weigh 5–10 grams of honey into a platinum dish, add a few drops of pure olive oil to prevent spattering, and heat carefully until swelling ceases and then ignite at a temperature not above dull redness until a white ash is obtained.

34 SOLUBLE ASH.—OFFICIAL.

Proceed as directed under **14**.

35 ALKALINITY OF THE SOLUBLE ASH.—OFFICIAL.

Proceed as directed under **15**.

POLARIZATION.

36 *Direct Polarization.—Tentative.*

(**a**) *Immediate direct polarization.*—Transfer 26 grams of the honey to a 100 cc. flask with water, add 5 cc. of alumina cream, dilute to the mark with water at 20°C., filter and polarize immediately in a 200 mm. tube.

(**b**) *Constant direct polarization.*—Pour the solution from the tube used in reading (**a**) back into the flask, stopper and allow to stand for 24 hours. At the end of this time again polarize the solution at 20°C. in a 200 mm. tube.

(**c**) *Birotation.*—The difference between (**a**) and (**b**) gives the birotation.

(**d**) *Direct polarization at 87°C.*—Polarize the solution, obtained as directed under (**b**), at 87°C. in a jacketed 200 mm. tube.

37 *Invert Polarization.—Tentative.*

(**a**) *At 20°C.*—Invert 50 cc. of the solution obtained in **36** as directed under **VII**, **14** or **16**, and polarize at 20°C. in a 200 mm. tube.

(**b**) *At 87°C.*—Polarize the solution, obtained as directed under (**a**), at 87°C. in a 200 mm. jacketed tube.

38 REDUCING SUGARS.—OFFICIAL.

Dilute 10 cc. of the solution, used for direct polarization, **36**, to 250 cc. and determine reducing sugars in 25 cc. of this solution by one of the methods given under **VII**, **25**, **35**, **38** or **55**, respectively. Calculate the result to per cent of invert sugar.

39 SUCROSE.—OFFICIAL.

Proceed as directed under **VII**, **18**. Determine reducing sugars after inversion by diluting 10 cc. of the solution obtained in **37** with a small amount of water, neutralizing with sodium carbonate and making up to 250 cc. with water. Employ 50 cc. of this solution for the determination, using the same method as in **38**.

40 LEVULOSE.—TENTATIVE.

Multiply the direct reading at 87°C., **36** (**d**), by 1.0315 and subtract the product from the constant direct polarization at 20°C., **36** (**b**); divide the difference by 2.3919 to obtain the grams of levulose in a normal weight of the honey. From this figure calculate the per cent of levulose in the original sample.

41 DEXTROSE.—TENTATIVE.

Subtract the per cent of levulose, obtained in **40**, from the per cent of invert sugar, found in **38**, to obtain the approximate per cent of dextrose.

The dextrose can be determined more accurately by multiplying the per cent of levulose, as found in **40**, by the factor 0.915, which gives its dextrose equivalent in copper reducing power. Subtract this figure from that of the reducing sugars, **38**, calculated as dextrose, to obtain the percentage of dextrose in the sample. (Owing to the difference in the reducing powers of different sugars, the sum of the dextrose thus found and the levulose as obtained in **40** will be greater than the amount of invert sugar obtained in **38**.)

42 DEXTRIN (APPROXIMATE).—TENTATIVE.

Transfer 8 grams of the sample (4 grams in the case of dark colored honey-dew honey) to a 100 cc. flask (using not more than 4 cc. of water) by allowing the sample to drain from the weighing dish into the flask and then dissolving the residue in 2 cc. of water. After adding this solution to the contents of the flask, rinse the weighing dish with two 1 cc. portions of water to which a little alcohol is added subsequently. Fill the flask to the mark with absolute alcohol, shaking constantly. Set the flask aside until the dextrin has collected on the sides and bottom and the liquid is clear. Decant the clear liquid through a filter paper and wash the residue in the flask with 10 cc. of 95 per cent alcohol, pouring the washings through the same filter. Dissolve the dextrin in the flask with boiling water and filter through the filter paper already used, receiving the filtrate in a tared dish, prepared as directed under **4**. Rinse the flask and wash the filter a number of times with small portions of hot water, evaporate on a water bath and dry to constant weight in vacuo at 70°C.

After determining the weight of the alcohol precipitate, dissolve the latter in water and make up to definite volume, using 50 cc. of water for each 0.5 gram of precipitate or part thereof.

Determine reducing sugars in the solution both before and after inversion as directed under **VII**, **18**, expressing the results as invert sugar. Calculate sucrose from the results thus obtained and subtract the sum of the reducing sugars before inversion and sucrose from the weight of the total alcohol precipitate to obtain the weight of the dextrin.

43 FREE ACID.—OFFICIAL.

Dissolve 10 grams of the honey in water and titrate with N/10 sodium hydroxid solution, using phenolphthalein as an indicator. Express the results in terms of cc. of N/10 sodium hydroxid required to neutralize 100 grams of the sample.

44 GLUCOSE.—TENTATIVE.

Qualitative test.—Dilute the honey with water in the proportion of 1 to 1, then add a few cc. of iodin solution (1 gram of iodin, 3 grams of potassium iodid, 50 cc. of water). In the presence of glucose the solution turns red or violet, the depth and character of the color depending upon the quality and nature of the glucose employed. A blank test with a pure honey of about the same color should be made in order to secure an accurate color comparison. Should the honey be dark and the percentage of glucose very small, precipitate the dextrin which may be present by adding several volumes of 95 per cent alcohol. Allow to stand until the precipitate settles (do not filter), decant the liquid, dissolve the residue of dextrins in hot water, cool and apply the above test to this solution. A negative result is not proof of the absence of glucose, as some glucose, especially of high conversion, does not give any reaction with iodin[7].

Quantitative test.—An approximate determination can be made by Browne's formula as follows: Multiply the difference in the polarizations of the invert solution at 20°C. and 87°C. by 77 and divide this product by the percentage of invert sugar after inversion found in the sample. Multiply the quotient by 100 and divide the product by 26.7 to obtain the percentage of honey in the sample; 100 per cent minus the per cent of honey gives the percentage of glucose.

COMMERCIAL INVERT SUGAR[8].

Resorcin test[9].—*Tentative.*

45 REAGENT.

Resorcin solution.—Dissolve 1 gram of resorcin in 100 cc. of hydrochloric acid, sp. gr. 1.19.

46 MANIPULATION.

Introduce 10 cc. of a 50 per cent honey solution into a test tube and add 5 cc. of ether. Shake gently and allow to stand for some time until the ether layer is clear. Transfer 2 cc. of this clear ether solution to a small test tube and add a large drop of the resorcin solution. Shake and note the color immediately. In the presence of artificial invert sugar, the resorcin assumes immediately an orange-red color turning to dark red.

Anilin Chlorid Test[10].—*Tentative.*

47 REAGENT.

Anilin chlorid solution.—To 100 cc. of C. P. anilin add 30 cc. of 25 per cent hydrochloric acid.

48 MANIPULATION.

Introduce 5 grams of the honey into a porcelain dish and add 2.5 cc. of the anilin reagent. A bright red color indicates the presence of commercial invert sugar.

49 DIASTASE[11].—TENTATIVE.

Mix 1 part of honey with 2 parts of sterile water. Treat 10 cc. of this solution with 1 cc. of 1 per cent soluble starch solution and digest at 45°C. for an hour. At the end of this time test the mixture with 1 cc. of iodin solution (1 gram of iodin, 2 grams of potassium iodid, 300 cc. of water). Treat another 10 cc. portion of the honey solution, mixed with 1 cc. of the soluble starch solution, without heating to 45°C., with the reagent and compare the colors produced. If the original honey had not been heated sufficiently to kill the diastase, an olive-green or brown coloration will be produced in the mixture that has been heated at 45°C. Heated or artificial honey becomes blue.

MAPLE PRODUCTS.

50 PREPARATION OF SAMPLE.—OFFICIAL.

(**a**) *Maple sirup.*—Determine the moisture by the method given under **51** (**a**). If the moisture is less than 35 per cent, and there is some mineral sediment, pour the clear sirup into a beaker, washing the sediment also into the beaker with water. Then concentrate the sirup by boiling to a moisture content of about 35 per cent (b. p. 104°C). Set aside until cool, or preferably let the covered material stand overnight, and pour off the clear liquid for the analytical work. Where no sediment is present the sample is ready for analysis after careful mixing. Where sugar has crystallized out, warm to dissolve the sugar before starting the analysis. It is desirable, in order to compare results upon different samples, to reduce all results other than moisture to a dry substance basis as determined in the clear sirup.

(**b**) *Maple sugar, maple cream, maple wax, etc.*—Determine moisture, by the method given under **51** (**b**), in the sample in its original condition, after thoroughly mixing, if semi-plastic, or after rubbing up in a mortar representative portions of the product, if solid. For all other analytical determinations use a solution prepared as follows: Weigh roughly 100 grams of the product into a beaker and dissolve by boiling with 200 cc. of water. Decant the resulting sirup while hot through a muslin filter, concentrate by boiling to a moisture content of 35 per cent (b. p. 104°C.), cool, or preferably let the covered material stand overnight, set aside until clear, and use this clear sirup for analysis. It is desirable, in order to compare results upon different samples, that all results except moisture be expressed upon a dry basis.

51 MOISTURE.—OFFICIAL.

(**a**) *Maple sirup.*—Proceed as directed under **32** or **9**.

(**b**) *Maple sugar, maple cream, etc.*—Proceed as directed under **32**.

52 POLARIZATION.—OFFICIAL.

(**a**) *Direct at 20°C.*—Proceed as directed under **VII, 14**.

(**b**) *Invert at 20°C.*—Proceed as directed under **VII, 14**.

(**c**) *Invert at 87°C.*—Proceed as directed under **22**.

53 REDUCING SUGARS AS INVERT SUGAR.—OFFICIAL.

(**a**) *Before inversion.*—Proceed as directed under **VII, 25**, using an aliquot of the solution used for direct polarization, **52** (**a**), and only neutral lead acetate for clarification.

(**b**) *After inversion.*—Proceed as directed under **VII, 25**, using an aliquot of the solution used for the invert polarization, **52** (**b**), and only neutral lead acetate for clarification.

SUCROSE.

54 *By Polarization.—Official.*

Proceed as directed under **VII, 14** or **16**.

55 *By Reducing Sugars Before and After Inversion.—Official.*

Proceed as directed under **VII, 18**.

56 COMMERCIAL GLUCOSE.—OFFICIAL.

Proceed as directed under **22**.

57 TOTAL ASH.—OFFICIAL.

Proceed as directed under **10**.

58 SOLUBLE AND INSOLUBLE ASH.—OFFICIAL.

Proceed as directed under **14**.

59 ALKALINITY OF THE SOLUBLE ASH.—OFFICIAL.

Proceed as directed under **15**.

60 ALKALINITY OF THE INSOLUBLE ASH.—OFFICIAL.

Proceed as directed under **16**.

LEAD NUMBER (WINTON).—TENTATIVE.

61 REAGENTS.

Standard basic lead acetate solution.—Boil 430 grams of neutral lead acetate and 130 grams of litharge, for 30 minutes, or boil 560 grams of Horne's dry basic lead acetate with 1 liter of water, cool, allow to settle and dilute the supernatant liquid to 1.25 sp. gr. To a measured amount of this solution add 4 volumes of water and filter if not perfectly clear. The solution should be standardized each time a set of determinations is made

If the directions for preparing the basic lead acetate are not carried out carefully, the use of Horne's dry basic lead acetate is preferable.

62 DETERMINATION OF LEAD IN THE BLANK.

Transfer 25 cc. of the standard basic lead acetate to a 100 cc. flask, add a few drops of acetic acid, and make up to the mark with water. Shake and determine lead sulphate in 10 cc. of the solution as directed under **63**. The use of the acid is imperative in this case to keep the lead in solution, when diluted with water.

63 DETERMINATION.

Transfer 25 grams of the sample to a 100 cc. flask by means of water. Add 25 cc. of the standard basic lead acetate and shake, fill to the mark, shake and allow to stand for at least 3 hours before filtering. Pipette 10 cc. of the clear filtrate into a 250 cc. beaker, add 40 cc. of water and 1 cc. of concentrated sulphuric acid, shake and add 100 cc. of 95 per cent alcohol. Allow to stand overnight, filter on a tared Gooch, wash with 95 per cent alcohol, dry in a water oven and ignite in a muffle or over a Bunsen burner, applying the heat gradually at first and avoiding a reducing flame. Cool and weigh. Subtract the weight of lead sulphate so found from the weight of lead sulphate found in the blank, **62**, and multiply by the factor 27.33. The use of this factor gives the lead number directly without the various calculations otherwise required.

64 MALIC-ACID VALUE (COWLES[13]).—TENTATIVE.

Weigh 6.7 grams of the sample into a 200 cc. beaker, add 5 cc. of water, then 2 cc. of a 10 per cent calcium acetate solution and stir. Add gradually, and with constant stirring, 100 cc. of 95 per cent alcohol, and agitate the solution until the precipitate settles, or let stand until the supernatant liquid is clear. Filter off the precipitate and wash with 75 cc. of 85 per cent alcohol. Dry the filter paper and ignite in a platinum dish. Add 10 cc. of N/10 hydrochloric acid and warm gently until all the lime dissolves.

Cool and titrate back with N/10 sodium hydroxid, using methyl orange as an indicator. The difference in cc., divided by 10, represents the malic acid value of the sample. Previous to use the reagents should be tested by a blank determination and any necessary corrections applied.

65 METALS.—TENTATIVE.

Proceed as directed under **XI.**

SUGAR HOUSE PRODUCTS.

SUCROSE IN BEETS.

66 *Alcohol Extraction Method*[13].—*Tentative.*

Weigh 26 grams of the beet pulp and transfer to a 100 cc. flask with about 50 cc. of 90 per cent alcohol and 3–5 cc. of basic lead acetate solution. Connect a reflux condenser to the flask and place on a boiling water bath for 10–15 minutes. Then pour the whole into a Soxhlet extractor, washing out the flask with fresh portions of 90 per cent alcohol. Connect the same 100 cc. flask to the extractor, and fit the latter with a return condenser. Add 90 per cent alcohol until the siphon is started and the flask is about three-fourths full. Place the flask in a covered water bath kept at a heat that will allow the alcohol to boil freely. Continue the extraction for 1–4 hours, or until a test of the alcohol in the extractor gives no color with alpha-naphthol solution when tested as follows: Introduce into a test tube a few drops of the alcohol coming from the extractor, add 4 or 5 drops of a 20 per cent alcoholic alpha-naphthol solution and 2 cc. of water. Shake well, tip the tube, and allow 2–5 cc. of colorless concentrated sulphuric acid to flow down the side of the tube; then hold the tube upright and, if sucrose is present, a color varying from a faint to a deep violet will be noted at the junction of the two liquids. On shaking, the whole solution becomes a blue violet color. This test is suitable for this work, but it must be remembered that other substances besides sucrose give this color reaction.

Remove the flask from the water bath, transfer the contents to a 100 cc. graduated flask, cool to the standard temperature, dilute to the mark with 90 per cent alcohol, shake and filter, keeping the funnel covered with a watch glass. Polarize in a 200 mm. tube.

Avoid evaporation and changes of temperature and also use a minimum amount of basic acetate for clarification, 3 cc. rather than 5 cc. By digesting the beet pulp with the alcohol before extraction, the time of extraction is greatly shortened, the pulp becomes thoroughly impregnated with the alcohol and all the air is removed, resulting in a good extraction of the whole material. If the pulp is fine and tends to clog the siphon, alcohol-washed cotton may be used as a plug in the extractor before adding the beet pulp and a fine mesh screen may be placed over the pulp to keep the whole compact in the extractor.

67 *Hot Water Digestion Method I*[14].—*Tentative.*

Weigh 52 grams of the beet cuttings and transfer them with water to a wide-mouthed flask graduated to a content of 201.2 cc.; add 5–10 cc. of basic lead acetate solution, fill the flask to the mark with hot water and shake. Immerse the flask in a water bath at 80°C. and rotate at intervals. Add water from time to time so that at the end of the heating (about 30 minutes) the water in the flask is a little above the mark. Remove the flask from the water bath and allow it to cool to standard temperature. Add sufficient concentrated acetic acid to make the solution very slightly acid (generally less than 0.5 cc.) and a few drops of ether to break the foam. Make up to the mark, mix thoroughly, filter and polarize in a 200 mm. tube.

The fineness of the pulp governs the time of heating. Add enough water at the start and maintain this volume during the extraction, so that not more than 5 cc. of water will be necessary to complete the volume after cooling. The proportion of pulp to water must not be increased beyond the prescribed amount, for when smaller proportions of water to pulp are used and then a large quantity of water is added at the last to make up to volume, the sugar does not become equally diffused and the results are too low. Differences of over 1 per cent in sugar content may be caused by lack of care in this particular.

68 *Hot Water Digestion Method II*[15].—*Tentative.*

There are needed nickel-plated sheet iron vessels, 11 cm. high, 6 cm. body diameter, and 4 cm. mouth diameter, also stoppers covered with tin foil to fit.

Weigh 26 grams of the beet pulp on a watch glass (small enough to go into the neck of the beaker) and transfer to the metal beaker, add 177 cc. of dilute basic lead acetate solution (5 parts of basic lead acetate solution (sp. gr. 1.25) to 100 parts of water), shake and stopper lightly. Submerge the beaker in a water bath at 75°-80°C. for 30 minutes, shaking intermittently. When all the air has been expelled (generally after 5 minutes), tighten the stopper. After 30 minutes, shake, cool to standard temperature, filter, add a drop of acetic acid to the filtrate and polarize in a 400 mm. tube. The reading is the per cent of sugar in the beet pulp.

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IX. FOOD PRESERVATIVES.

SALICYLIC ACID.

1 PREPARATION OF SAMPLE.—OFFICIAL.

(**a**) *Non-alcoholic liquids.*—Many liquids may be extracted directly as described in **2** or **4** without further treatment. If gums or mucilaginous substances are present which cause troublesome emulsions during extraction, pipette 100 cc. into a 250 cc. volumetric flask, add about 5 grams of sodium chlorid, shake until the latter is dissolved, make up to the mark with alcohol, shake vigorously, allow the mixture to stand for 10 minutes with occasional shaking, filter through a dry folded filter and treat an aliquot of the filtrate as directed under (**b**).

(**b**) *Alcoholic liquids.*—Make 200 cc. of the sample alkaline with sodium hydroxid solution, using litmus as an indicator, and evaporate on a steam bath to about one-third its original volume. Dilute to the original volume with water and filter, if necessary, through a dry filter.

(**c**) *Solid or semi-solid substances.*—Grind the sample and mix thoroughly. Transfer a convenient quantity (50–200 grams according to the consistency of the sample) to a 500 cc. volumetric flask, add sufficient water to make a volume of about 400 cc., shake until the mixture becomes uniform, add 2–5 grams of calcium chlorid, shake until the latter is dissolved, render distinctly alkaline with sodium hydroxid solution, using litmus as an indicator, fill to the mark with water, shake thoroughly, allow to stand for at least 2 hours, shaking frequently, and filter through a large folded filter.

DETECTION AND ESTIMATION.

2 *Ferric Chlorid Test.—Qualitative.—Official.*

Introduce 50 cc. of the sample or an equivalent amount of an aqueous extract, prepared as directed under **1**, into a separatory funnel, add one-tenth its volume of dilute hydrochloric acid (1 to 3) and extract with 50–100 cc. of ether. If the mixture emulsifies, add 10–15 cc. of petroleum ether (b. p. below 60°C.) and shake. If this treatment fails to break the emulsion whirl the mixture in a centrifuge, or allow it to stand until a considerable portion of the aqueous layer has separated, run off the latter, shake vigorously and again allow to separate. Wash the ether layer with two 5 cc. portions of water, evaporate the greater portion of the ether in a porcelain dish on a steam bath, allow the remainder to evaporate spontaneously and add a drop of 0.5 per cent ferric chlorid solution. A violet color indicates salicylic acid.

If coloring matter or other interfering substances are present in the residue left after evaporation of the ether, purify the salicylic acid by one of the following methods:

(**a**) Dissolve the residue from the ether extract, obtained as directed above, in about 25 cc. of ether, transfer the latter to a separatory funnel and shake with an equal quantity of water, made distinctly alkaline with several drops of ammonium hydroxid. Allow to separate, filter the aqueous layer through a wet filter into a porcelain dish, evaporate almost to dryness and test the residue as directed above.

(**b**) Dry the residue from the ether extract, obtained as directed above, in a desiccator over sulphuric acid and extract with several 10 cc. portions of carbon disulphid or petroleum ether (b. p. below 60°C.), rubbing the contents of the dish with a glass rod and filtering the successive portions of the solvent through a dry paper into a second porcelain dish. Evaporate the greater portion of the solvent on a steam bath, allow the remainder to evaporate spontaneously and test the residue as directed above.

(**c**) Transfer the residue from the ether extract, obtained as directed above, to a small porcelain crucible by means of a few cc. of ether and allow the solvent to evaporate spontaneously. Cut a hole in a piece of asbestos board sufficiently large to admit about two-thirds of the crucible, cover the latter with a small, round-bottomed flask filled with cold water and heat over a small Bunsen flame until any salicylic acid present has sublimed and condensed upon the bottom of the flask. Test the sublimate as directed above.

3 *Jorissen Test*[1].—*Qualitative.*—*Official.*

Dissolve the residue from the ether extract, obtained as directed under **2**, or, in case impurities are present, the purified material obtained as directed under **2** (**a**), (**b**) or (**c**) in a little hot water. Cool 10 cc. of the solution in a test tube, add 4 or 5 drops of 10 per cent potassium nitrite solution, 4 or 5 drops of 50 per cent acetic acid and 1 drop of 10 per cent cupric sulphate solution, mix thoroughly and heat to boiling. Boil for half a minute and allow to stand for 1–2 minutes. In the presence of salicylic acid a blood red color will develop.

Colorimetric Method.—*Quantitative.*—*Official.*

4 EXTRACTION.

Pipette a convenient portion of the sample (100 cc. or a volume representing not less than 20 grams of the original sample) or a solution, prepared as in **1**, into a separatory funnel, make the solution neutral to litmus with dilute hydrochloric acid (1 to 3) and add an excess of concentrated hydrochloric acid equivalent to 2 cc. of acid for each 100 cc. of solution. Extract with 4 separate portions of ether, using for each extraction a volume of ether equivalent to half the volume of the aqueous layer. If an emulsion forms on shaking, this may usually be broken by adding a little (one-fifth the volume of the ether layer) petroleum ether (b. p. below 60°C.) and shaking again or by centrifugalizing. If a small amount of emulsion still persists, allow it to remain with the aqueous layer, where it is frequently broken during the next extraction. If an emulsion remains after the fourth extraction, separate it from the clear ether and the clear aqueous layer and extract it separately with 2 or 3 small portions of ether. Combine the ether extracts, wash with one-tenth their volume of water, allow the layers to separate and reject the aqueous layer. Wash in this way until the aqueous layer after separation yields a yellow color upon the addition of methyl orange and 2 drops of N/10 sodium hydroxid. Distil slowly the greater part of the ether, transfer the remainder to a porcelain dish and allow it to evaporate spontaneously. If there are no interfering substances present, proceed as directed under **5**. If such interfering substances are present, purify the residue by one of the following methods:

(**a**) Dry thoroughly the residue in vacuo over sulphuric acid and extract with 10 portions of 10–15 cc. each of carbon disulphid or petroleum ether (b. p. below 60°C.), rub the contents of the dish with a glass rod and filter the successive portions of the solvent through a dry filter into a porcelain dish. Test the extracted residue with a drop of ferric alum solution and, if it gives a reaction for salicylic acid, dissolve it in water and re-extract with ether, proceeding as directed above. Distil the greater portion of the carbon disulphid or petroleum ether and allow the remainder to evaporate spontaneously. Proceed as directed under **5**.

(**b**) Dissolve the residue in 40–50 cc. of ether. Transfer the ether solution to a separatory funnel and extract with 3 successive 15 cc. portions of 1 per cent ammonium hydroxid. (If fat is known to be present in the original ether extract, extract the latter directly with 4 portions of the ammonium hydroxid instead of 3.) Combine the alkaline aqueous extracts, acidify, again extract with ether and wash the combined ether extracts as directed above. Distil slowly the greater portion of the ether, allow the remainder to evaporate spontaneously and proceed as directed under **5**.

5 DETERMINATION

Dissolve the residue, obtained in **4**, in a small amount of hot water and, after cooling, dilute to a definite volume (usually 50–100 cc.), dependent on the amount of salicylic acid present. If the solution is not clear, filter through a dry filter. Dilute aliquots of the solution and treat with a few drops of 0.5 per cent ferric chlorid solution or 2 per cent ferric alum solution.

The ferric alum solution should be boiled until a precipitate appears, allowed to settle, and filtered. The acidity of the solution is slightly increased in this manner, but it remains clear for a considerable time and the turbidity caused by its dilution with water is much less and does not appear so soon as when the unboiled solution is used. This turbidity interferes with the exact matching of the color.

Compare the colors developed with that obtained when a standard salicylic acid solution (containing 1 mg. of salicylic acid in 50 cc.) is similarly treated, using Nessler tubes or a colorimeter. In either case, and especially with ferric chlorid, avoid an excess of the reagent, although an excess of 0.5 cc. of 2 per cent ferric alum solution may be added to 50 cc. of the comparison solution of salicylic acid without impairing the results.

BENZOIC ACID.

PREPARATION OF SAMPLE.—OFFICIAL.

6 *General Method.*

If the sample is solid or semi-solid, grind it, and mix thoroughly. Transfer about 150 grams to a 500 cc. graduated flask, add enough pulverized sodium chlorid to saturate the water in the sample, render alkaline with sodium hydroxid solution or milk of lime and dilute to the mark with saturated salt solution. Allow to stand for at least 2 hours, with frequent shaking, and filter. If the sample contains large amounts of matter precipitable by salt solution, it is advisable to follow a method similar to that given under **7 (d)**. When alcohol is present, follow the method given under **7 (c)**. When large amounts of fats are present, make the filtrate alkaline and extract before proceeding as directed under **11**.

7 *Special Methods.*

(a) *Ketchup.*—Saturate the water in 150 grams of ketchup by adding 15 grams of pulverized sodium chlorid. Transfer the mixture to a 500 cc. graduated flask, rinsing with about 150 cc. of saturated sodium chlorid solution. Make slightly alkaline to litmus paper with strong sodium hydroxid solution and fill to the mark with saturated salt solution. Allow to stand for at least 2 hours, shaking frequently. Squeeze through a heavy muslin bag and then filter through a large folded filter.

(b) *Jellies, jams, preserves and marmalades.*—Dissolve 150 grams of the sample in about 300 cc. of saturated salt solution. Add 15 grams of pulverized sodium chlorid. Make alkaline to litmus paper with milk of lime. Transfer to a 500 cc. graduated flask and dilute to the mark with saturated salt solution. Allow to stand for at least 2 hours, shaking frequently, centrifugalize if necessary and filter through a large folded filter.

(c) *Cider containing alcohol, and similar products.*—Make 250 cc. of the sample alkaline to litmus paper with sodium hydroxid solution and evaporate on a steam bath to about 100 cc. Transfer the sample to a 250 cc. graduated flask, add 30 grams of pulverized sodium chlorid and shake until dissolved. Dilute to the original volume, 250 cc., with saturated salt solution, allow to stand for at least 2 hours, shaking frequently, and filter through a folded filter.

(d) *Salted or dried fish.*—Wash 50 grams of the ground sample into a 500 cc. graduated flask with water. Make slightly alkaline to litmus paper with strong sodium hydroxid solution and dilute to the mark with water. Allow to stand for at least 2 hours, shaking frequently, and then filter through a folded filter. Pipette accurately as large a portion of the filtrate as possible (at least 300 cc.) into a second 500 cc. flask. Add 30 grams of the pulverized sodium chlorid for each 100 cc. of solution. Shake until the salt has dissolved and dilute to the mark with saturated salt solution. Mix thoroughly and filter off the precipitated protein matter on a folded filter.

8 DETECTION AND ESTIMATION.

Extract benzoic acid as directed under **2** or **4**. If benzoic acid is present in considerable quantity, it will crystallize from the ether in shining leaflets having a characteristic odor on heating. Dissolve the residue in hot water divide into 2 portions and test according to **9** or **10**. The residue may also be purified as directed under **2** (**c**) and the melting point determined.

9 *Ferric Chlorid Test.—Qualitative.—Official.*

Make the solution from **8** alkaline with ammonium hydroxid, expel the excess of ammonia by evaporation, dissolve the residue in water and add a few drops of a neutral 0.5 per cent ferric chlorid solution. A brownish precipitate of ferric benzoate indicates the presence of benzoic acid.

10 *Modified Mohler Test*[2]*.—Qualitative.—Official.*

(The presence of phenolphthalein interferes with this test.)

Add to the water solution, prepared as described under **8**, 1–3 cc. of N/3 sodium hydroxid and evaporate to dryness. To the residue, add 5–10 drops of concentrated sulphuric acid and a small crystal of potassium nitrate. Heat for 10 minutes in a glycerol bath at 120°–130°C., or for 20 minutes in a boiling water bath. The temperature must not exceed 130°C. After cooling add 1 cc. of water and make distinctly ammoniacal; boil the solution to decompose any ammonium nitrite which may have been formed. Cool and add a drop of fresh, colorless ammonium sulphid, without allowing the layers to mix. A red-brown ring indicates benzoic acid. On mixing, the color diffuses through the whole liquid and, on heating, finally changes to greenish yellow. This differentiates benzoic acid from salicylic acid or cinnamic acid. The last two form colored compounds, which are not destroyed by heating.

11 *Quantitative Method.—Official.*

Pipette a convenient portion (100–200 cc.) of the filtrate, obtained in **6** or **7**, into a separatory funnel. Neutralize the solution to litmus paper with hydrochloric acid (1 to 3) and add an excess of 5 cc. of the same acid. In the case of salted fish a precipitation of protein matter usually occurs on acidifying, but the precipitate does not interfere with the extraction. Extract carefully with chloroform, using successive portions of 70, 50, 40, and 30 cc. To avoid the formation of an emulsion, shake cautiously each time. The chloroform layer usually separates readily after standing a few minutes. If an emulsion forms, break it: (1) by stirring the chloroform layer with a glass rod; (2) by drawing it off into a second funnel and giving 1 or 2 sharp shakes from one end of the funnel to the other; or (3) by centrifugalizing for a few minutes. As this is a progressive extraction, draw off carefully as much of the clear chloroform solution as possible after each extraction, but do not draw off any of the emulsion with the chloroform layer. If this precaution is taken, the chloroform extract need not be washed.

Transfer the combined chloroform extracts to a porcelain evaporating dish, rinse the container several times with a few cc. of chloroform and evaporate to dryness at room temperature in a current of air dried over calcium chlorid.

The extract may also be transferred from the separatory funnel to a 300 cc. Erlenmeyer flask, rinsing the separatory funnel 3 times with 5–10 cc. portions of chloroform. Distil very carefully to about one-fourth the original volume, keeping the temperature down so that the chloroform comes over in drops, not in a steady stream. Then transfer the residue to a porcelain evaporating dish, rinsing the flask 3 times with 5–10 cc. portions of chloroform, and allow to evaporate to dryness spontaneously.

Dry the residue overnight (or until no odor of acetic acid can be detected if the product is a ketchup) in a desiccator containing sulphuric acid. Dissolve the residue of benzoic acid in 30–50 cc. of neutral alcohol, add about one-fourth this volume of water, 1 or 2 drops of phenolphthalein, and titrate with N/20 sodium hydroxid (1 cc. is equivalent to 0.0072 gram of anhydrous sodium benzoate).

SACCHARIN.

12

Qualitative Test.—Official.

Extract with ether (after maceration and exhaustion with water, if necessary), as directed in **14**. Allow the ether extract to evaporate spontaneously and note the taste of the residue. The presence of saccharin, to the extent of 20 mg. per liter, is indicated by a sweet taste. Confirm by heating with sodium hydroxid, as described below, and detecting the salicylic acid formed thereby. A sweet taste, suggesting the presence of a trace of saccharin, has been obtained frequently in saccharin-free wines, due to the so-called "false saccharin".

Acidify 50 cc. of a non-alcoholic liquid food or the aqueous extract of 50 grams of a solid or semi-solid, prepared as directed in **13**, and extract with ether as directed in **14**. Dissolve the residue, remaining after evaporation of the ether, in a little hot water and test a small portion of this solution for salicylic acid as directed under **2** or **3**. Dilute the remainder of the solution to about 10 cc., and add 2 cc. of sulphuric acid (1 to 3). Heat to boiling and add a slight excess of 5 per cent potassium permanganate solution, drop by drop; partly cool the solution, dissolve a piece of sodium hydroxid in it and filter the mixture into a silver dish (silver crucible lids are well adapted to the purpose); evaporate to dryness and heat for 20 minutes at 210°–215°C. Dissolve the residue in water, acidify with hydrochloric acid and test the ether extract for salicylic acid as directed under **2** or **3**. By this method all the so-called "false saccharin" and the salicylic acid naturally present (also added salicylic acid when not present in too large an amount) are destroyed, while 5 mg. of saccharin per liter are detected with certainty.

Quantitative Method.—Offiical.

13

PREPARATION OF SAMPLE.

(**a**) *Fruit juices, sirups and other non-alcoholic liquids.*—Transfer 100–200 grams of the sample to a 250 cc. volumetric flask by means of a little water, dilute to about 200 cc. with water, add 5 cc. of glacial acetic acid, mix, add a slight excess of 20 per cent neutral lead acetate solution, mix thoroughly, dilute to the mark with water, again mix thoroughly and filter through a folded filter.

(b) *Alcoholic liquids.*—Heat 100–200 cc. of the liquid on a steam bath to remove alcohol, this being accomplished in most cases by evaporating to one-half the original volume. In the case of heavy sirups the liquid should be diluted with an equal volume of water before beginning the evaporation. After the alcohol has been removed transfer to a 250 cc. volumetric flask and proceed from this point as directed in (a).

(c) *Solid or semi-solid preparations.*—Transfer 50–75 grams of the sample to a 250 cc. volumetric flask by means of a little hot water and add sufficient nearly boiling water to make the volume about 200 cc. Allow the mixture to stand for 2 hours, shaking occasionally. Then add 5 cc. of glacial acetic acid, mix thoroughly, add a slight excess of 20 per cent neutral lead acetate solution, dilute to the mark with cold water, mix and allow to stand for 20 minutes. Filter through a folded filter.

14 DETERMINATION.

Transfer 150 cc. of the filtrate (obtained by one of the above methods) to a separatory funnel, add 15 cc. of concentrated hydrochloric acid and extract 3 times with 80 cc. portions of ether, shaking the separatory funnel for 2 minutes each time. Wash the combined ether extracts once with 5 cc. of water, remove the ether by distillation and transfer the residue to a platinum crucible by means of a little ether; or if substances difficultly soluble in ether are present use alternate small portions of water and ether. Evaporate the ether on a steam bath, add to the residue 2–3 cc. (or enough to make the mixture strongly alkaline) of a 10 per cent sodium carbonate solution, rotate so that all the saccharin is brought in contact with the solution and evaporate to dryness on a steam bath. To the dry residue in the crucible add 4 grams of a mixture of equal parts of anhydrous sodium and potassium carbonates, heat gently at first and then to complete fusion for 30 minutes over an alcohol or other sulphur-free flame. The fusion may be conducted with a gas flame by closely fitting the crucible into a hole cut into a piece of heavy asbestos board so that one-third of the crucible projects above the asbestos, and heating the lower portion of the crucible by means of a large Bunsen or Meker burner. Cool, dissolve the melt in water, add about 5 cc. of bromin water. acidify with hydrochloric acid, filter, wash the paper with a little water, dilute the filtrate and washings to about 200 cc., heat to boiling and slowly add an excess of barium chlorid solution. Allow to stand overnight, collect the barium sulphate on a filter (or a platinum Gooch crucible), wash until free from chlorids, dry, ignite, cool and weigh. Correct the result thus obtained for any sulphur present in the fusion mixture as found by a blank determination. Calculate the equivalent amount of saccharin by multiplying the corrected weight of barium sulphate by 0.7845.

Instead of the mixed sodium and potassium carbonates, 3–4 grams of sodium peroxid may be employed for the fusion. In this case a nickel crucible must be used, and the time of fusion may be reduced to 5 minutes.

The separation of a little lead chlorid during the extractions does not interfere with the accuracy of the method.

BORIC ACID AND BORATES.

15 *Qualitative Test*[1].—*Official.*

Preliminary test.—Immerse a strip of turmeric paper in the sample acidified with hydrochloric acid in the proportion of 7 cc. of concentrated acid to each 100 cc. of sample and allow the paper to dry spontaneously. If borax or boric acid is present, the paper will acquire a peculiar red color, changed by ammonium hydroxid to a dark blue-green but restored by acid. Solid or pasty samples may be heated with enough water to make them sufficiently fluid, concentrated hydrochloric acid added in about the proportion of 1 to 13 and the liquid tested in the same way.

Confirmatory test.—Make about 25 grams of the sample decidedly alkaline with lime water and evaporate to dryness on a water bath. Ignite the residue to destroy organic matter. Digest with about 15 cc. of water, add concentrated hydrochloric acid, drop by drop, until the ignited residue is dissolved and then add 1 cc. in excess. Saturate a piece of turmeric paper with the solution, and allow it to dry without the aid of heat. In the presence of borax or boric acid, the color change will be the same as given above.

16 *Quantitative Method*[4].—*Official.*

Make 10–100 grams of the sample (depending upon the nature of the sample and the amount of boric acid present) distinctly alkaline with sodium hydroxid solution and evaporate to dryness in a platinum dish. Ignite the residue until organic matter is destroyed, avoiding an intense red heat, cool, digest with about 20 cc. of hot water and add hydrochloric acid, drop by drop, until the reaction is distinctly acid. Filter into a 100 cc. flask, and wash with a little hot water, the volume of the filtrate not to exceed 50–60 cc. Return the filter containing any unburned carbon to the platinum dish, make alkaline by wetting thoroughly with lime water, dry on a steam bath and ignite to a white ash. Dissolve the ash in a few cc. of dilute hydrochloric acid and add to the liquid in the 100 cc. flask, rinsing the dish with a few cc. of water. To the combined solutions, add 0.5–5 grams of calcium chlorid and a few drops of phenolphthalein, then 10 per cent sodium hydroxid solution until a permanent light pink color is produced and finally dilute to the mark with lime water. Mix and filter through a dry filter. To 50 cc. of the filtrate add N/1 sulphuric acid until the pink color disappears, then add methyl orange, and continue the addition of the acid until the yellow color is changed to pink. Boil for about 1 minute to expel carbon dioxid. Cool, and carefully add N/5 sodium hydroxid until the liquid assumes a yellow tinge, avoiding an excess of the alkali. All the boric acid is now in a free state with no uncombined sulphuric acid present. Add an equal volume of neutral glycerol and a little phenolphthalein. Titrate with N/5 sodium hydroxid until a permanent pink color is produced. About 10 grams of mannitol may be substituted for the glycerol in this determination. At the end of the titration add an additional 2 grams and continue the titration if the pink color is discharged. Repeat the alternate addition of mannitol and alkali until a permanent end point is reached.

One cc. of N/5 sodium hydroxid is equivalent to 0.0124 gram of boric acid.

FORMALDEHYDE.

17 PREPARATION OF SAMPLE.—OFFICIAL.

If the sample is solid or semi-solid, macerate 200–300 grams of the material with about 100 cc. of water in a mortar. Transfer to a short-necked, 500–800 cc. copper or glass distillation flask and make distinctly acid with phosphoric acid, connect with a condenser and distil 40–50 cc. In the case of highly colored liquids, the same method of preparation should be employed.

18 *Phenylhydrazin Hydrochlorid test*[5].—*Official.*

In the case of milk and other liquids shake with an equal volume of strong alcohol, filter from any insoluble matter and use the filtrate. In the case of meats and fats, extract the formaldehyde with alcohol and use the filtrate. In the case of fat, heat the mixture above the melting point of the fat to insure thorough extraction.

Mix 5 cc. of the distillate, as prepared under **17**, or of an alcoholic solution or extract, obtained as directed above, with 0.03 gram of phenylhydrazin hydrochlorid and 4 or

5 drops of a 1 per cent ferric chlorid solution. Add slowly and with agitation, in a bath of cold water to prevent heating the liquid, 1–2 cc. of concentrated sulphuric acid. Dissolve the precipitate by the addition of either concentrated sulphuric acid (keeping the mixture cool) or alcohol. In the presence of formaldehyde a red color develops.

This method gives reliable reactions for formaldehyde in solutions of formaldehyde varying from 1 part in 50,000 to 1 part in 150,000. Acetaldehyde and benzaldehyde give no reaction when treated by this method and do not interfere with the reaction given by formaldehyde.

19 *Hehner Method*[6].—*Official.*

Mix about 5 cc. of the distillate, obtained in **17**, with an equal volume of pure milk, or a 1–2 per cent solution of egg albumen, in a test tube and underlay with strong commercial sulphuric acid without mixing. A violet or blue color at the junction of the 2 liquids indicates formaldehyde. This color is given only in the presence of a trace of ferric chlorid or other oxidizing agent. As pointed out by Hehner, milk may be treated directly by this method and gives positive tests in the presence of 1 or more parts of formaldehyde per 10,000, Some other articles of food rich in proteins, for example, egg albumen, give the reaction in the presence of water without the addition of milk.

20 *Leach Method.—Official.*

Mix about 5 cc. of the distillate, obtained under **17**, with an equal volume of pure milk in a porcelain casserole and add about 10 cc. of concentrated hydrochloric acid, containing 1 cc. of 10 per cent ferric chlorid solution, to each 500 cc. of acid. Heat to 80°–90°C. directly over the gas flame, rotating the casserole to break up the curd. A violet coloration indicates formaldehyde.

21 *Phenylhydrazin Hydrochlorid and Sodium Nitroprussid Test*[7].—*Official.*

This method may be applied directly to liquid foods, to an aqueous or alcoholic extract of solid foods, or to the distillate prepared as directed under **17**. In the case of milk, apply the method directly. In the case of meat, comminute the sample, extract with 2 volumes of hot water and employ the expressed liquid for the test. Heat fats above their melting point with 10 cc. of alcohol, shake thoroughly, cool, filter through a moistened filter and use the filtrate for the test.

Dissolve a lump of phenylhydrazin hydrochlorid about the size of a pea in 3–5 cc. of the liquid to be tested, add 2–4 drops (not more) of a 5–10 per cent sodium nitroprussid solution and 8–12 drops of an approximately 12 per cent sodium hydroxid solution. If formaldehyde is present, a green or blue color develops depending upon the amount. When formaldehyde is present to the extent of more than 1 part in 70,000–80,000 in the solution tested, a distinct green or bluish green coloration is obtained. In more dilute solutions the green tint becomes less marked and a yellow tinge tending toward greenish brown develops.

With this method acetaldehyde and benzaldehyde give a color varying from red to brown, according to the strength of the solution. A reaction may therefore be obtained with these aldehydes similar to that obtained with formaldehyde in solutions more dilute than 1 part in 70,000. The presence of acetaldehyde or benzaldehyde together with formaldehyde gives a yellowish or yellowish green tinge. The reaction for formaldehyde may therefore be masked by the presence of other aldehydes, but is characteristic when a clear green color is obtained.

22 *Phenylhydrazin Hydrochlorid and Potassium Ferricyanid Test*[7].—*Official.*

Proceed as directed under **21**, substituting a solution of potassium ferricyanid for the sodium nitroprussid. Formaldehyde gives a red color. Alcoholic extracts from foods must be diluted with water to prevent the precipitation of potassium ferricyanid. The test is not applicable in the presence of the coloring matter of blood.

23 *Phenylhydrazin Hydrochlorid and Ferric Chlorid Test*[7].—*Official.*

Treat 15 cc. of milk or other liquid food or of the distillate, prepared as directed under **17**, with 1 cc. of a dilute phenylhydrazin hydrochlorid solution, then with a few drops of dilute ferric chlorid solution and finally with concentrated hydrochloric acid. The presence of formaldehyde is indicated by the formation of a red color, which changes after some time to orange yellow.

Milk may be examined directly by this method, but more delicate tests may be obtained from the distillate from milk or from milk serum. Acetaldehyde or benzaldehyde does not interfere with the reaction.

24 *Phloroglucol Test*[8].—*Official.*

To 10 cc. of milk or other liquid food under examination in a test tube add, by means of a pipette, 2 cc. of phloroglucol reagent (1 gram of phloroglucol, 20 grams of sodium hydroxid and water to make 100 cc.), placing the end of the pipette on the bottom of the tube in such a manner that the reagent will form a separate layer.

If formaldehyde be present, a bright red coloration (not purple) forms at the zone of contact. This solution gives a yellow color in the presence of some aldehydes and, if it is used for the detection of aldehyde formed by the oxidation of methyl alcohol after the destruction of acetaldehyde with hydrogen peroxid, an orange yellow color will slowly appear when an insufficient amount of hydrogen peroxid has been employed. On the other hand, if the excess of hydrogen peroxid is not fully destroyed before the use of this reagent, a purple color develops slowly. The clear, red color given by the use of this reagent forms quickly and, in the presence of but a small amount of formaldehyde, fades rapidly.

FLUORIDS.

25 *Method I*[9].—*Official.*

Thoroughly mix the sample and boil 150 cc. (in the case of solid foods an aqueous extract may be employed provided the fluorids are in a soluble form). Add to the boiling liquid 5 cc. of 10 per cent potassium sulphate solution and 10 cc. of 10 per cent barium acetate solution. Collect the precipitate in a compact mass (a centrifuge may be used advantageously) and wash upon a small filter. Transfer to a platinum crucible and ignite.

Dip a carefully cleaned glass plate, while hot, in a mixture of equal parts of Carnaüba wax and paraffin and allow to cool. Make, with a sharp instrument, a distinctive mark through the wax, taking care not to scratch the surface of the glass.

Add a few drops of concentrated sulphuric acid to the residue in the crucible and cover with the waxed plate, having the mark nearly over the center and making sure that the edge of the crucible is in close contact with it. Keep the top surface of the plate cool by means of a suitable device and heat the crucible for an hour at as high a temperature as practicable without melting the wax (an electric stove gives the most satisfactory form of heat).

If fluorids be present, a distinct etching will be apparent on the glass where it was exposed.

26 *Method II.—Official.*

The preceding method may be varied by mixing a small amount of precipitated silica with the precipitated barium fluorid and applying the method for the detection of fluosilicates, under **28** or **29**.

This method is of value in the case of foods the ash of which contains a considerable amount of silica. Under these circumstances, concentrated sulphuric acid liberates silicon fluorid, which would escape detection under **25**.

FLUOBORATES AND FLUOSILICATES.

27 PREPARATION OF SAMPLE.—OFFICIAL.

Make about 200 grams of the sample alkaline with lime water, evaporate to dryness and incinerate. Extract the crude ash with water, to which sufficient acetic acid has been added to decompose carbonates, filter, ignite the insoluble portion, extract with dilute acetic acid and again filter. The insoluble portion now contains calcium silicate and fluorid, while the filtrate will contain all the boric acid present.

28 *Method I[10].—Official.*

Incinerate the filter, from **27**, containing the insoluble portion, mix with a little precipitated silica, transfer to a short test tube, attached to a small U-tube containing a few drops of water, and add 1–2 cc. of concentrated sulphuric acid. Keep the test tube in a beaker of water on the steam bath for 30–40 minutes. If any fluorin be present, the silicon fluorid generated will be decomposed by the water in the U-tube and will form a gelatinous deposit on the walls of the tube.

Next test the filtrate as directed under **15**. If both hydrofluoric and boric acids be present, it is probable that they are combined as borofluorid. If, however, silicon fluorid is detected and not boric acid, the operation should be repeated without the introduction of the silica, in which case the formation of the silicon skeleton is conclusive evidence of the presence of fluosilicate. In an ash containing an appreciable amount of silica, sulphuric acid will liberate silicon fluorid rather than hydrofluoric acid. The presence of a fluosilicate is indicated, therefore, and not the presence of a fluorid.

29 *Method II.—Official.*

Incinerate the filter, from **27**, containing the insoluble portion, in a platinum crucible, mix with a little precipitated silica and add 1 cc. of concentrated sulphuric acid. Cover the crucible with a watch glass, from the underside of which a drop of water is suspended, and heat for an hour at 70°–80°C., keeping the watch glass cooled. The silicon fluorid which is formed is decomposed by the water, leaving a gelatinous deposit of silica and etching a ring at the periphery of the drop of water. Test the filtrate for boric acid as directed under **15**.

SULPHUROUS ACID.

30 *Qualitative Test[11].—Official.*

Add some sulphur-free zinc and several cc. of hydrochloric acid to about 25 grams of the sample (with the addition of water, if necessary) in a 200 cc. Erlenmeyer flask. In the presence of sulphites, hydrogen sulphid will be generated and may be detected with lead acetate paper. Traces of metallic sulphids are occasionally present in vegetables, and will give the same reaction as sulphites under the conditions of the above test. Positive results obtained by this method should be verified by the distillation method under **31**.

It is always advisable to make the quantitative determination of sulphites, owing to the danger that the test may be due to traces of sulphids. A trace is not to be considered sufficient indication of the presence of sulphur dioxid either as a bleaching agent or as a preservative.

TOTAL SULPHUROUS ACID.

31 *Method I. (Distillation Method.)—Official.*

Distil 20–100 grams of the sample (adding recently boiled water if necessary) in a current of carbon dioxid, after the addition of about 5 cc. of a 20 per cent glacial phosphoric acid solution, until 150 cc. have passed over. Collect the distillate in about 100 cc. of nearly saturated bromin water, allowing the end of the condenser to dip below the surface. The method and apparatus may be simplified without material loss in accuracy by omitting the current of carbon dioxid, adding 10 cc. of phosphoric acid instead of 5 cc., and dropping into the distillation flask, immediately before attaching the condenser, a piece of sodium bicarbonate weighing not more than 1 gram. The carbon dioxid liberated is not sufficient to entirely expel the air from the apparatus, but will prevent oxidation to a large extent. When the distillation is finished, boil off the excess of bromin, dilute the solution to about 250 cc., add 5 cc. of hydrochloric acid (1 to 3), heat to boiling and precipitate the sulphuric acid with 10 per cent barium chlorid solution. Boil for a few minutes longer, allow to stand overnight in a warm place, filter on a weighed Gooch crucible, wash with hot water, ignite at a dull red heat and weigh as barium sulphate.

32 *Method II. (Direct Titration Method.)—Official.*

In the examination of wine, fairly accurate results may be obtained by the following method:

Place 25 cc. of 5.6 per cent potassium hydroxid solution in a 200 cc. flask. Introduce 50 cc. of the sample, mix with the potassium hydroxid solution and allow the mixture to stand for 15 minutes with occasional agitation. Add 10 cc. of sulphuric acid (1 to 3) and a few cc. of starch solution, and titrate the mixture with N/50 iodin solution. Introduce the iodin solution as rapidly as possible and continue the addition until the blue color persists for several minutes. One cc. of N/50 iodin is equivalent to 0.00064 gram of sulphur dioxod.

33 FREE SULPHUROUS ACID.—OFFICIAL.

(Especially Adapted to Wine.)

Treat 50 cc. of the sample in a 200 cc. flask with about 5 cc. of sulphuric acid (1 to 3), add about 0.5 gram of sodium carbonate to expel the air and titrate the sulphurous acid with N/50 iodin, as directed under **32**.

BETA-NAPHTHOL.

34 *Qualitative Test.—Tentative.*

In a separatory funnel extract 200 cc. of the sample, or of its aqueous extract, prepared as directed under **1** (**c**), with 10 cc. of chloroform. To the chloroform extract in a test tube add a few drops of alcoholic potash, and place in a boiling water bath for 2 minutes. The presence of beta-naphthol is indicated by the formation of a deep blue color, which changes to green and then to yellow.

ABRASTOL.

35 *Sinibaldi Method*[12].—*Tentative.*

Make 50 cc. of the sample alkaline with a few drops of ammonium hydroxid and extract with 10 cc. of amyl alcohol, adding ethyl alcohol if an emulsion is formed. Decant the amyl alcohol, filter if turbid, and evaporate to dryness. Add to the residue 2 cc. of nitric acid (1 to 1), heat on the water bath until half of the liquid is evaporated and transfer to a test tube with the addition of 1 cc. of water. Add about 0.2 gram of ferrous sulphate and an excess of ammonium hydroxid, drop by drop, with constant shaking. If the resultant precipitate is of a reddish color, dissolve it in a few drops of sulphuric acid and add ferrous sulphate and ammonium hydroxid as before. As soon as a dark colored or greenish precipitate is obtained, introduce 5 cc. of alcohol, dissolve the precipitate in sulphuric acid, shake well and filter. In the absence of abrastol this method gives a colorless or light yellow liquid, while a red color is produced in the presence of 0.01 gram of abrastol.

36 *Sanglé-Ferrière Method*[13].—*Tentative.*

Boil 200 cc. of the sample with 8 cc. of concentrated hydrochloric acid for an hour in a flask fitted with a reflux condenser. Abrastol is thus converted into betanaphthol and is detected as directed under **34**.

SUCROL OR DULCIN.

37 *Morpurgo Method*[14].—*Tentative.*

Evaporate about 100 cc. of the sample, or of the aqueous extract prepared as directed under **1** (**c**) and neutralized with acetic acid, to a sirupy consistency after the addition of about 5 grams of lead carbonate, and extract the residue several times with 90 per cent alcohol. Evaporate the alcoholic extract to dryness, extract the residue with ether and allow the ether to evaporate spontaneoulsy in a porcelain dish. Add 2 or 3 drops each of phenol and concentrated sulphuric acid and heat for about 5 minutes on the water bath, cool, transfer to a test tube and overlay with ammonium hydroxid or sodium hydroxid solution with the least possible mixing. The presence of dulcin is indicated by the formation of a blue color at the zone of contact.

38 *Jorissen Method*[15].—*Tentative.*

Suspend the residue from the ether extract, obtained as directed above, in about 5 cc. of water; add 2–4 cc. of an approximately 10 per cent solution of mercuric nitrate and heat for 5–10 minutes on the water bath. In the presence of sucrol a violet blue color is formed, which is changed to a deep violet on the addition of lead peroxid.

FORMIC ACID[16].—OFFICIAL.

39 REAGENTS.

(**a**) *Sodium acetate solution.*—Dissolve 50 grams of dry sodium acetate in sufficient water to make 100 cc. and filter.

(**b**) *Mercuric chlorid reagent.*—Dissolve 100 grams of mercuric chlorid and 150 grams of sodium chlorid in sufficient water to make 1 liter and filter.

(**c**) *Tartaric acid.*

(**d**) *Barium carbonate.*

40 APPARATUS.

The apparatus required (Fig. 4) consists of a steam generator (*S*), a 300 cc. flask (*A*) in which the sample is placed, a 500 cc. flask (*B*), containing a suspension of barium carbonate, a spray trap (*T*), a condenser, and a 1 liter graduated flask (*C*). The tip of the tube (*D*), leading into (*B*), consists of a bulb containing a number of small holes to break the vapor into small bubbles.

41 DETERMINATION.

For thin liquids like fruit juices, use 50 cc. For heavy liquids and semi-solids like sirups and jams, use 50 grams diluted with 50 cc. of water. Place the sample in the flask (*A*), add 1 gram of tartaric acid, and connect as shown in Fig. 4, the flask (*B*) having been charged previously with a suspension of 2 grams of barium carbonate in 100 cc. of water. If much acetic acid is present, sufficient barium carbonate must be used so that at least 1 gram remains at the end of the operation. Heat the contents of flasks (*A*) and (*B*) to boiling and distil with steam from the generator (*S*), the vapor passing first through the sample in flask (*A*), then through the boiling suspension of barium carbonate in (*B*), after which it is condensed, and measured in the graduated flask (*C*). Continue the distillation until 1 liter of distillate is collected, maintaining the volume of the liquids in the flasks (*A*) and (*B*) as nearly constant as possible by heating with small Bunsen flames, and avoiding charring of the sample in the flask (*A*). After 1 liter of distillate has been collected, disconnect the apparatus and filter the contents of flask (*B*) while hot, washing the barium carbonate with a little hot water. The filtrate and washings should now measure about 150 cc. If not they should be boiled down to that volume. Then add 10 cc. of the sodium acetate, 2 cc. of 10 per

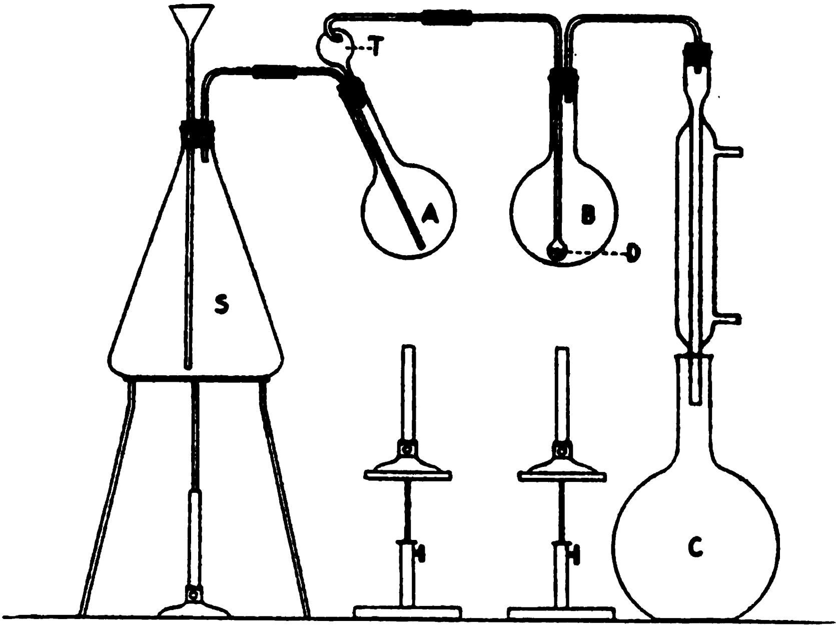

FIG. 4. APPARATUS FOR DETERMINATION OF FORMIC ACID.

cent hydrochloric acid and 25 cc. of the mercuric chlorid reagent. Mix thoroughly and immerse the container in a boiling water bath or steam bath for 2 hours. Then filter on a tared Gooch crucible, wash the precipitate thoroughly with cold water and finally with a little alcohol. Dry in a boiling water oven for 30 minutes, cool, weigh and calculate the weight of formic acid present by multiplying the weight of the precipitate by 0.0975. If the weight of mercurous chlorid obtained exceeds 1.5 grams, the determination must be repeated, using more mercuric chlorid reagent or a smaller amount of sample. A blank test should be conducted with each new lot of reagents employed in the reduction, using 150 cc. of water, 1 cc. of 10 per cent barium chlorid solution, 2 cc. of 10 per cent hydrochloric acid, 10 cc. of the sodium acetate, and 25 cc. of the mercuric chlorid reagent, heating the mixture in a boiling water bath or steam bath for 2 hours. The weight of mercurous chlorid obtained in this blank test must be deducted from that obtained in the regular determination.

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X. COLORING MATTERS IN FOODS.—TENTATIVE.

(An italicized number, following immediately the name of a dye, is the number by which that dye is designated in "A Systematic Survey of the Organic Colouring Matters", 1904, by Arthur Green, based on the German of Schultz and Julius.)

1 PIGMENTS.

The insoluble pigments, ultramarine, lampblack, etc., are most commonly used as facings and may be separated by washing the sample with water and allowing the washings to settle. The particles of coloring matter can be identified by microscopic examination and by treatment of the residue or purified coloring matter with chemical reagents. A large proportion of the common pigments other than lakes, such as the yellow, brown and red ochres and umbers, are derivatives of the heavy metals and contain iron, manganese etc. Others, such as various green and blue compounds, including the green chlorophyll derivatives, contain copper. These pigments may be identified by the usual tests for the respective metals. The analytical properties of the insoluble coloring matters are described in various standard works, some of which are listed in the bibliography, especially "Farbstofftabellen", by Schultz[1].

SOLUBLE COLORING MATTERS AND THEIR LAKES.

COAL TAR DYES.

2 *Wool dyeing test*[2].

(a) *Wines, fruit juices, distilled liquors, flavoring extracts, vinegars, beers, sirups, non-alcoholic beverages and similar products.*—Dilute 20–200 cc. of the sample with 1–3 volumes of water and boil or heat on the steam bath with a small piece of white woolen cloth (nun's veiling). When the mixture contains much alcohol, heat until most of the alcohol has been removed; in other cases, take out the wool after 5–15 minutes and rinse with water. Then treat the liquid with 3 or 4 drops of concentrated hydrochloric acid for each 100 cc. and warm again for 10–20 minutes with a clean piece of wool. The basic dyes go on the fiber best from neutral or faintly ammoniacal solutions and, if present, will appear on the first piece of wool. Acid colors dye from neutral solutions but more readily from those containing free acid. If the wool takes up any considerable amount of coloring matter in either case, the presence of coal tar dyes is indicated. The lichen colors[3] (Archil, Cudbear, Litmus) go readily on wool, however, and many other natural colors, such as Turmeric, will dye the fiber, if present in considerable amount. On the other hand, a few coal tar dyes, especially Auramine O and Naphthol Green B, are quite unstable and, if present in small amounts, may give no distinct dyeing. Acid dyes are much more frequently used than basic dyes and in most cases may be removed from wool without much decomposition by "stripping" the latter with dilute ammonia[4]. By the action of the alkali, many natural colors are destroyed, while others remain for the most part on the fiber. If the behavior with wool in neutral and acid solutions indicates the presence of acid dyes, rinse the colored cloth thoroughly with water, cover with 2 per cent ammonium hydroxid solution in a casserole, boil for a few minutes, remove the cloth and squeeze out the adhering liquid. Boil the ammoniacal solution to remove the excess of ammonia, drop in a piece of clean, wet wool, make distinctly but not strongly acid with hydrochloric acid and boil again. If acid coal tar dyes are present, they will usually give a fairly clean, bright dyeing on the second piece of wool. A further purification may be carried out by repeating the stripping and re-dyeing, though generally accompanied by corresponding loss of dye.

(**b**) *Candies and similar colored sugar products.*—Dissolve about 20 grams of the sample in 100 cc. of water and treat the solution as directed under (**a**). When the coloring matter is on the surface of the candy, pour off the solution before the colorless inner portion has dissolved.

(**c**) *Jams and jellies.*—Boil a mixture of 10–20 grams of the sample and 100 cc. of water with wool in neutral and also in acid solution as directed under (**a**). For thick jams it is usually better, though less easy, first to extract the coloring substances by treating the product as directed under (**d**).

(**d**) *Canned and preserved fruits and vegetables, sausage casings, smoked fish, coffee, spices, etc.*—Macerate 20–200 grams of the sample with 4–5 times its weight of 80 per cent alcohol. After standing a few hours pour off the solvent as completely as possible and repeat the extraction, using 70 per cent alcohol containing about 1 per cent of ammonia. (1) Examine separately the filtered alcoholic extracts as directed under (**a**); or, (2) Boil the ammoniacal solution until practically neutral, complete the neutralization with acetic acid, add the neutral 80 per cent alcohol extract, continue the evaporation until most of the alcohol is removed and boil with wool as directed under (**a**).

(**e**) *Cocoa and chocolate products.*—Treat cocoa as directed under (**d**). The alcoholic extract will contain a large amount of natural coloring matter and several dyeings and strippings may be necessary to get rid of this in order to show the presence of coal tar dyes.

Chocolate may be treated similarly but the following procedure is preferable: Wash 20–200 grams of the well-divided sample with gasoline on a filter until most of the fat has been removed; if the gasoline is colored, reserve for the examination of oil-soluble dyes as directed under **3**. Remove most of the adherent solvent from the residue by evaporation or pressure between layers of absorbent paper and digest with alcohol as directed under (**d**).

Coal tar dyes may also be detected in chocolate and cocoa products by mixing samples directly with 3–4 times their weight of hot water and immediately boiling the magma with wool, as directed under (**a**). Because of the presence of large amounts of fatty and protein materials, this method is not very satisfactory.

(**f**) *Cereal products.*—Proceed as directed under (**d**), in most cases working with a large amount of the sample, 200–500 grams, and a relatively smaller amount of alcohol. Where tests are to be made only for the acid dyes, the extraction with neutral 80 per cent alcohol may be omitted advantageously.

3 OIL-SOLUBLE DYES[5].

Prepare an alcoholic solution of the oil-soluble dye by one of the following methods, which are to be applied to the oil or fat obtained by extraction with ether or gasoline if the nature of the substance requires it:

(**a**) Shake the oil or melted fat with an equal volume of 90 per cent alcohol. The alcohol after separation will contain Aniline Yellow, Butter Yellow, Aminoazotoluene and Auramine, if present.

(**b**) Saponify 20–200 grams of the oil or fat with alcoholic potash and, after removal of most of the alcohol on the steam bath, extract the soap with ether or gasoline. Most of the common dyes are removed by this treatment, though the digestion with strong alkali may cause some decomposition and make the extraction rather troublesome.

(**c**) Dilute 20–200 grams of the oil or melted fat with 1–2 volumes of gasoline and shake out successively with 2–4 per cent potassium or sodium hydroxid solution, 12–15 per cent hydrochloric acid, and phosphoric-sulphuric acid mixture, prepared by mixing 85 per cent phosphoric acid with about 10–20 per cent by volume of concentrated sulphuric acid.

The dilute alkali extracts Sudan G and Annatto. The dilute hydrochloric acid extracts Aniline Yellow (*7*), Aminoazotoluene, and Butter Yellow (*16*), the first two forming orange-red, the latter cherry-red solutions in this solvent. The phosphoric acid mixture is necessary for the extraction of Sudan I (*11*), Sudan II (*49*), Sudan III (*143*), and the homologue of the last, Sudan IV. Benzeneazobeta-naphthylamin and homologues also come in this group, though they readily undergo chemical changes in the strongly acid mixtures. The procedure is not very suitable in the presence of Auramine but this dye is seldom found in oils. Neutralize the alkaline and dilute hydrochloric acid solutions; dilute the phosphoric acid mixture and partially neutralize, cooling the liquid during this operation; and extract the dyes by shaking with ether or gasoline.

For the direct dyeing test use the alcoholic solution obtained as directed in (**a**). Evaporate to dryness the ether or gasoline solutions, obtained as directed in (**b**) and (**c**), and dissolve the residue in 10–20 cc. of strong alcohol. To the alcoholic solution add some strands of white silk and a little water and evaporate on the steam bath until the alcohol has been removed or until the dye is taken up by the silk. The dyeing test is sometimes unsatisfactory, and in all cases a small portion of the alcoholic solution should be tested by treating with an equal volume of concentrated hydrochloric acid and stannous chlorid solution. The common oil-soluble coal tar dyes are rendered more red or blue by the acid and are decolorized by the reducing agent. Most of the natural coloring matters become slightly paler with the acid and are little changed by the stannous chlorid solution.

SEPARATION OF COLORING MATTERS IN PURE CONDITION BY MEANS OF IMMISCIBLE SOLVENTS[6].

4 PREPARATION OF SAMPLE.

(**a**) *Water-soluble colors.*—Proceed as directed under **2**, omitting the fixation of the color on wool, and obtain an aqueous solution as free as practicable from suspended matter, alcohol, acids, alkalies and salts.

(**b**) *Water-insoluble lakes.*—If the sample is in solid form, treat the well-divided material with sufficient water to form a paste. Liquids require no preparation except the removal of any alcohol which may be present.

5 MIXTURES OF ORANGE I, ERYTHROSINE, INDIGO CARMINE, AMARANTH, TARTRAZINE, NAPHTHOL YELLOW S, PONCEAU 3R AND LIGHT GREEN S F YELLOWISH.

The use of immiscible solvents for the separation of mixtures of coloring matters usually involves a systematic fractionation, since many of the dyes used do not differ very greatly in their solubilities in the various solvents. When it seems probable that only the 8 water-soluble coal tar dyes permitted under the Federal Food and Drugs Act[7] are present, the following abridged procedure may be used for their separation. For this procedure the concentration of the dye solution should lie preferably between 0.01 and 0.05 per cent. The solutions obtained in the examination of colored food products practically never require further dilution. but with commercial food colors care must be taken that the concentration is not too high. Treat the sample, prepared as directed in **4**, with one-half its volume of concentrated hydrochloric acid and extract a few times with amyl alcohol. The use of the centrifuge is sometimes necessary to separate the layers. Designate the residual aqueous layer as *A*. Combine the amyl alcohol extracts and wash with 4–5 portions of N/4 hydrochloric acid or until this solvent extracts very little color. These washings will contain any Indigo Carmine, Amaranth and Tartrazine present, the Indigo Carmine being removed in somewhat larger proportion in the first washings than the other two. With ordinary concentration very little Ponceau will be removed. Designate these combined washings as *B*.

6 ORANGE I AND ERYTHROSINE.

Measure, if necessary, the amyl alcohol extract from which some of the colors have been removed, under **5**, then (1) Dilute with an equal volume of petroleum ether or low boiling gasoline, and again wash several times with N/4 hydrochloric acid to extract Ponceau 3R and Naphthol Yellow S; or, (2) Without dilution with gasoline, wash with 5 per cent salt solution until these two dyes are extracted. Designate these washings as *C*. The Ponceau and Yellow having been removed, the amyl alcohol, containing an equal volume of gasoline, is washed a few times with water to extract Orange I. This dye having been removed, shake the solution, although the latter may appear almost colorless, with very dilute sodium hydroxid solution to remove Erythrosine. If considerable Orange I is present, some of it may contaminate the washings containing the Ponceau 3R and Naphthol Yellow S, especially when these have been separated by means of N/4 hydrochloric acid after the addition of gasoline.

7 INDIGO CARMINE, AMARANTH AND TARTRAZINE.

The presence of two or all three of these dyes is usually indicated by the appearance of the N/4 hydrochloric acid washings, *B*, under **5**. Evaporate the combined N/4 hydrochloric acid washings to dryness to remove the excess of hydrochloric acid and dissolved amyl alcohol. Dissolve the residue in water, divide the solution and identify the constituent colors in the portions. To a portion of the slightly acidified solution add a few decigrams of urea, warm and add 1 or 2 drops of sodium nitrite solution. Indigo Carmine is converted into the pale yellow isatin sulphonate while the other dyes are but little affected. The isatin compound is not ordinarily present in sufficient concentration to tint the solution, but it differs from Tartrazine also in being much less readily extracted by amyl alcohol from strong acid solutions (less than one-half from 4N acid). The solution now contains the Amaranth or Tartrazine, or both, practically unaffected. Amaranth is much more quickly attacked by reducing agents than Tartrazine. Treat the solution, which should be neutral or faintly acid (in the presence of sodium carbonate the reduction of the tartrazine takes place still more slowly), at room temperature with a dilute solution of sodium hydrosulphite ($Na_2S_2O_4$), adding the latter very carefully, drop by drop, and allowing sufficient time after the addition of each drop for the reduction to take place. When the color shows that the Amaranth has been destroyed completely, shake the mixture at once with air to oxidize the slight excess of hydrosulphite before it can react further on the Tartrazine.

To separate the Indigo Carmine heat to boiling another portion of the solution, which should be neutral or faintly acid, and add dilute sodium hydrosulphite solution, drop by drop, until all the dyes are reduced. On shaking with air the Indigo Carmine is quickly re-formed.

8 NAPHTHOL YELLOW S AND PONCEAU 3R.

Treat the N/4 acid solution or the salt solution, *C*, under **6**, as the case may be, containing the Ponceau and Naphthol Yellow S, with enough hydrochloric acid to make it about 2N and shake out a few times with washed ethyl acetate[8]. Remove the Yellow from the combined ethyl acetate extracts by shaking with water. Naphthol Yellow S is almost colorless in strongly acid solutions, and its absence in washings, etc., must never be assumed until these have been made alkaline. Finally separate the Ponceau 3R from the acid solution by shaking with amyl alcohol, and then wash out the dye from this extract with a few small portions of water. If, in the case of mixtures containing Orange I, the washings of the ethyl acetate, which should contain only Naphthol Yellow S, become more red upon the addition of alkalies, combine them, then (1) Make N/4 with hydrochloric acid and remove the contaminating Orange by shaking with amyl alcohol-gasoline mixture (1 to 1); or, (2) Treat the combined washings with one-fifth their volume of concentrated hydrochloric acid, extract the dyes by shaking once with amyl alcohol, and remove the Yellow by washing with several portions of 5 per cent salt solution.

9 LIGHT GREEN S F YELLOWISH.

The original mixture, *A*, under **5**, from which the above mentioned seven colors have been removed by adding acid and shaking out with amyl alcohol, may still contain Light Green S F Yellowish, which will be colorless or nearly so in the acid solution. To separate this dye treat the mixture with strong ammonia or potassium hydroxid solution until slightly alkaline, and neutralize with acetic acid. Any Green present will now be apparent by the color of the mixture. Extract the color by shaking with a few small portions of dichlorhydrin. Wash the dichlorhydrin extract with a little water, then dilute with several volumes of benzene or carbon tetrachlorid, and remove the dye by shaking with water.

When coal tar dyes other than the eight mentioned above are present, the solutions obtained in this procedure will be found to contain a coloring matter which does not correspond exactly in properties to one of the dyes named above. When coal tar dyes other than these eight are present, reference should be made to the larger works[9].

COAL TAR DYES IN GENERAL.

10 BASIC DYES.

Most basic dyes may be separated from mixtures by making alkaline with sodium hydroxid and shaking with ether[10]. Use the sample, prepared as in **4**, for this purpose. Separate the ether layer, which may or may not be colored, and shake with 2–5 per cent acetic acid, which will take up any dye present, forming a colored solution. Although the common basic colors may undergo some alteration by this treatment, it can be used for the qualitative detection and separation of Methyl Violet B (*451*), Magenta (*448*), Bismarck Brown (*197*), Malachite Green (*427*), and Rhodamine B (*504*). With care Auramine (*425*) also may be separated in this way, though it is quickly decomposed on standing in alkaline solution.

11 ACID DYES.

The following short procedure is often convenient for the examination of mixtures of acid dyes: Make the sample, prepared as in **4**, the color concentration of which does not vary greatly from 0.01–0.05 per cent, strongly acid by adding one-half its volume of concentrated hydrochloric acid and shake with amyl alcohol. Separate the amyl alcohol solution and wash by shaking with successive portions of one-half its volume of water, reserving the portions in separate test tubes or beakers. Because of the acid dissolved in the amyl alcohol these washings will show a regular decrease in acidity and the coloring matters will appear *in maximum* amount in the different fractions according to their respective solubilities. Ponceau 6R (*108*) is washed out chiefly while the acidity is still high, N/1 or above. Amaranth (*107*), Brilliant Scarlet (*106*) and Tartrazine (*94*) appear when the washings have an acidity between N/1 and N/4; Orange G (*14*) and Soluble Blue (*480*) between N/2 and N/16; Palatine Scarlet (*53*), Ponceau 2R (*55*) and 3R (*56*), Naphthol Yellow S (*4*), Cochineal (*706*), Crystal Ponceau (*64*) and Azorubine A (*103*) between N/16 and N/256. When the acid is practically all removed, Orange I (*85*), Orange II (*86*) and Croceine Orange (*13*) begin to wash out, and less readily, Orange IV (*88*) and Metanil Yellow (*95*). Finally the unsulphonated coloring matters, such as Erythrosine G (*516*), Erythrosine (*517*) and the Rose Bengals (*520* and *523*) are removed very slowly by water or not at all when all traces of acid have been removed. Acid Yellow (*8*) and Brilliant Yellow S (*89*) are not very uniform in composition. They are partially taken up by amyl alcohol from acid solution and appear chiefly in the first washings. Indigo Carmine (*692*) behaves somewhat similarly.

IDENTIFICATION OF THE COAL TAR DYES[11].

12 GENERAL.

The most widely used tests for the identification of coal tar dyes refer to the changes produced with acids and alkalies. Other tests, based upon the behavior with reducing agents, followed perhaps by treatment with oxidants or by separation and identification of the reduction products[12], and tests based upon oxidation of the dye and treatment of the oxidation products[13], are generally applicable. Spectroscopic methods are also used[14].

13 COLOR CHANGES PRODUCED WITH ACIDS AND ALKALIES.

Transfer the separated coloring matter to wool (or to silk in the case of oil-soluble dyes) by boiling as directed under **2** (**a**) or **3**. Care should be taken that the final dyeing is made in a solution fairly free from foreign matter such as sugar or aromatic substances, which, adhering to the fiber, may modify the reaction. In most cases the amount of color available is small and should not be dyed on too large a piece of wool (or silk). Rinse the dyed fibre thoroughly in running water, dry, cut into small pieces and place separately in the depressions of a white porcelain spot plate. Moisten the pieces with the respective reagents employed. (For many coloring matters the hue upon treatment with acids or alkalies varies markedly with the concentration of the reagents and amount of dye present; therefore the unknown dye should be compared with dyeings of known colors of approximately the same dye concentration as shown by this appearance.)

The table under **14** shows the color changes produced by concentrated hydrochloric and sulphuric acids, 10 per cent sodium hydroxid and 12 per cent ammonium hydroxid solutions on wool dyed with 0.1–0.5 per cent of the respective coloring matters. Included also are the reactions of the oil-soluble colors, but these refer to dyeings on silk. The dyes are arranged approximately according to hue. Brown is classed with orange; black (gray), with violet.

14

TABLE 9.

Color reactions produced on dyed fibers by various reagents.

COLORING MATTER	NO.	HYDROCHLORIC ACID	SULPHURIC ACID	SODIUM HYDROXID	AMMONIUM HYDROXID
Rhodamine B	*504*	Orange	Yellow	Bluer	Bluer
Rose Bengal	*523*	Almost decolorized	Orange	No change	No change
Archil	*710*	Red	Dull brown	Violet	Violet
Magenta	*448*	Yellowish brown	Dull brown	Decolorized	Paler
Acid Magenta	*462*	Almost decolorized	Yellow	Decolorized	Decolorized
Palatine Red	*62*	Darker	Violet	Dull brown	Little change
Bordeaux B	*65*	Violet	Blue	Brown	Little change
Amaranth	*107*	Slightly darker	Violet to brownish	Dull brownish	Little change
Azorubine A	*103*	Little change	Violet	Red	Red
Erythrosine	*517*	Orange-yellow	Orange-yellow	No change	No change
Ponceau 6RB	*169*	Blue	Blue	Dull violet-red	Little change
Ponceau 6R	*108*	Violet-red	Violet	Brown	Orange-red
Crystal Ponceau	*64*	Violet-red	Violet	Dull brown	Little change
Ponceau 3R	*56*	Little change	Little change	Dull orange	Little change
Sudan III*	*143*	Violet, then brown	Green	Violet-red	Little change
Safranine	*584*	Greenish blue	Green	Red	Red
Brilliant Scarlet	*106*	Red	Violet-red	Yellowish brown	Orange-red
Ponceau 2R	*55*	Little change	Little change	Brownish yellow	No change
Palatine Scarlet	*53*	Darker	Violet-red	Brownish yellow	No change
Erythrosine G	*516*	Yellow-orange	Yellow-orange	No change	No change
Sudan II*	*49*	Red	Violet-red	Little change	No change
Sudan I*	*11*	Orange-red	Red	Redder	No change
Cochineal	*706*	Little change	Little change	Violet-red	Violet-red
Bismarck Brown	*197*	Redder, darker	Browner	Yellower	Yellower
Bismarck Brown R	*201*	Redder, darker	Browner	Yellower	Yellower
Orange I	*85*	Violet	Violet	Red, dark	Red, dark
Orange II	*86*	Red	Red	Dull red	No change
Croceine Orange	*13*	Orange-red	Orange	Slightly darker	No change
Orange G	*14*	Little change	Orange	Dull, brownish red	No change
Orthotolueneazo-beta-naphthylamine*		Red	Violet	Little change	No change
Benzeneazo beta naphthylamine		Red	Violet	Little change	No change
Sudan G*	*10*	Orange-yellow	Brownish yellow	Orange-yellow	No change
Butter Yellow*	*16*	Violet-red	Orange-yellow	No change	No change
Aniline Yellow*	*7*	Brownish-red	Orange-yellow	Little change	No change
Aminoazo-ortho-toluene*		Dull orange	Orange-yellow	Little change	No change

* Oil-soluble.

14 TABLE 9.—Concluded.

COLORING MATTER	NO.	HYDROCHLORIC ACID	SULPHURIC ACID	SODIUM HYDROXID	AMMONIUM HYDROXID
Fluoresceine	*510*	Little change	Little change	Green fluorescent	Green fluorescent
Metanil Yellow	*95*	Violet-red	Violet	No change	No change
Azoflavine	*92*	Violet-red	Violet-red	Dull brown	Little change
Acid Yellow	*8*	Red	Orange	Little change	No change
Brilliant Yellow S	*89*	Violet-red	Violet-red	Little change	Little change
Tartrazine	*94*	Slightly darker	Slightly darker	Little change	Little change
Naphthol Yellow S	*4*	Almost decolorized	Very pale, dull brown	No change	No change
Auramine	*425*	Decolorized	Almost decolorized	Decolorized	Paler
Turmeric	*707*	Red	Reddish brown	Orange	Orange
Quinoline Yellow	*667*	Slightly darker	Brownish yellow	Slightly paler	Little change
Naphthol Green B	*398*	Yellowish	Brownish yellow	No change	No change
Guinea Green B	*433*	Pale orange-yellow	Pale, dull yellow	Decolorized	Decolorized
Light Green SF Yellowish	*435*	Pale orange-yellow	Pale, dull yellow	Decolorized	Decolorized
Night Green 2B	*438*	Pale orange-yellow	Pale, dull yellow	Decolorized	Paler
Malachite Green	*427*	Almost decolorized	Almost decolorized	Decolorized	Decolorized
Erioglaucine A	*436*	Yellow	Pale, dull yellow or brown	Slightly darker	Little change
Patent Blue A	*442*	Pale orange-yellow	Pale or dull brown	Little change	Little change
Soluble Blue	*480*	Paler	Brown	Pale reddish	Almost decolorized
Indigo Carmine	*692*	Slightly darker	Slightly darker	Greenish yellow	Greenish blue
Formyl Violet	*468*	Pale orange-yellow	Pale, dull orange	Decolorized	Decolorized
Methyl Violet B	*451*	Yellowish	Yellowish	Decolorized	Almost deolorized
Nigrosine, soluble	*602*	Dull bluish	Dull greenish	Brownish red, paler	Pale reddish

15 SPECIAL TESTS FOR COAL TAR DYES PERMITTED[7] UNDER THE FEDERAL FOOD AND DRUGS ACT.

The dyes, given in **5**, are sufficiently characterized in most cases by the solubilities shown in their separation and by the color changes given by acids and alkalies on the dyed fiber. This is especially true with Amaranth, Tartrazine, and Orange I. By treatment with reducing agents such as stannous chlorid, titanous chlorid, zinc dust or sodium hydrosulphite in acid solution, Indigo Carmine, Amaranth, Tartrazine, Ponceau 3R and Orange I are decolorized. With Indigo Carmine the color returns on shaking with air, most readily on warming, or on the addition of oxidizing agents such as ferric chlorid or potassium persulphate. Excess of the reducing agents must of course be avoided. With the four last named dyes the color is not restored. Dilute solutions of

Light Green S. F. Yellowish, Naphthol Yellow S and Erythrosine become paler or colorless with acids so that the effects of acid reducing agents are not so readily apparent. Neutral solutions of Naphthol Yellow S are decolorized by sodium hydrosulphite and other reducing agents, the color not returning with air or oxidants. An evanescent deepening of the shade may take place immediately upon the addition of the hydrosulphite. Erythrosine and Light Green S F Yellowish become paler with sodium hydrosulphite, the color being partially restored upon the addition of potassium persulphate.

In hot solutions containing an excess of sodium tartrate, the dyes named are readily decolorized by titanium trichlorid[15]. In the case of Indigo Carmine, if the reducing agent has been added carefully and an excess avoided, the blue color readily returns on shaking with air. With Erythrosine and Light Green S F Yellowish the color is scarcely restored by air but on cooling and adding potassium persulphate returns imperfectly. The reduction products of the other dyes do not give colored solutions again on oxidation if a slight yellowish or brownish tint that may sometimes appear be disregarded.

Indigo Carmine is extracted in small proportions from slightly acid solutions by shaking with dichlorhydrin. Most of the other common bluish dyes are triphenylmethane derivatives and are relatively more soluble in this liquid than in the aqueous layer. A small portion (1 cc.) of the solution obtained in the separation, as described under 5, may be used directly.

Ponceau 3R gives in neutral or faintly acid solutions a bluish red, flocculent precipitate with barium chlorid or acetate, practically all of the dye being removed from solution. Some of the solution obtained in the separation, under 5, may be used in this test, first neutralizing the free hydrochloric acid with sodium acetate; or better, it may be evaporated to dryness on the steam bath to remove the acid and the residue taken up with a little water. The solution should contain 0.005 per cent or more of the dye.

Naphthol Yellow S, in solutions containing an excess of ammonia or sodium carbonate, becomes intensely rose-red on the addition of sodium hydrosulphite, the color gradually fading again as complete reduction takes place.

Erythrosine differs from most of the common dyes by containing iodin. To test for this, acidify the solution with sulphuric acid, shake with ether, separate the ether solution of the color and evaporate to dryness in a platinum dish after the addition of a few drops of sodium carbonate solution or sufficient to form the deep red sodium salt. Hold the dish containing the residue in the Bunsen flame until organic matter is destroyed, take up the residue with water, acidify with sulphuric acid and test for iodin in one of the usual ways, such as with chlorin water and carbon disulphid or tetrachlorid, or with starch paste and an oxidizing agent. It is useless to test for iodin with very small amounts of dye but in most cases sufficient coloring matter can be separated from the food product to give satisfactory results.

16 NATURAL COLORING MATTERS.

The natural coloring matters as a class show much less tendency to dye animal fiber than do the common synthetic colors. In many cases the crude products used contain a number of colored substances and a complete separation can scarcely be attempted. Most of the natural coloring matters, in dilute solution, are sensitive to alkalies, some to acids, hence such reagents must be used with care.

SEPARATION OF NATURAL COLORING MATTERS.

17 *Extraction with ether from neutral solutions.*

From neutral solutions ether extracts Carotin, Xanthophyll (the pigments found in leaves, fats and oils, egg yoke, carrots, etc.), the coloring matter of tomatoes and paprika and green Chlorophyll. The coloring matter remains in the ether solution on shaking with dilute sodium hydroxid solution or dilute hydrochloric acid, no apparent change taking place although chemically the substances may be altered more or less by this treatment.

18 *Extraction with ether from acid solutions.*

From slightly acid solutions ether extracts very readily and completely the coloring matter of Alkanet, Annatto, Turmeric, and the red dyewoods, Sandalwood, Camwood and Barwood. It extracts in large proportions the flavone coloring matters of Fustic, and of Persian Berries and Quercitron (after hydrolysis), as well as the coloring matter of Brazilwood and the green derivatives formed from Chlorophyll by alkaline treatment. It extracts in relatively small amount the coloring matters of Logwood, Archil, Saffron and Cochineal. The coloring matters of this group are readily removed from ether by shaking with alkaline solutions but in most cases rapidly undergo chemical change.

19 *Extraction with amyl alcohol from acid solutions.*

From slightly acid solutions amyl alcohol extracts largely the coloring matters of Logwood, Archil, Saffron and Cochineal. [From ammoniacal Cochineal (Carmine) the ordinary coloring matter is readily re-formed upon standing with hydrochloric acid.] Amyl alcohol extracts in relatively small proportions Caramel and the Anthocyans constituting the red coloring matter of the most common fruits.

IDENTIFICATION OF NATURAL COLORING MATTERS.

20 REAGENTS.

(**a**) *Hydrochloric acid.*—Sp. gr. 1.20.

(**b**) *10 per cent sodium or potassium hydroxid solution.*

(**c**) *Sodium hydrosulphite solution.*—A freshly prepared 5 per cent solution of "Blankite", sodiun hydrosulphite ($Na_2S_2O_4$).

(**d**) *0.5 per cent ferric chlorid solution.*—Freshly prepared but may be made by diluting a 10 per cent stock solution.

(**e**) *10 per cent potassium or ammonium alum solution.*

(**f**) *5 per cent uranium or sodium uranium acetate solution.*

(**g**) *Sulphuric acid.*—Sp. gr. 1.84.

21 PROCEDURE.

Relatively few good tests are known for the common natural colors. Some of their most useful analytical properties[16] are tabulated in **22**. In general these tests should be applied to the somewhat purified solutions of the coloring matter obtained as indicated in **17**, **18** or **19**.

The properties of pure preparations of the various natural coloring matters are described, for the most part, by Rupe[17], and by Perkin and Everest[18], reference being made in these works to the original literature. Properties of the Chlorophylls and Carotinoids are given by Willstätter and Stoll[19]. Those of the coloring matters of the Corn Flower, Rose, Pelargona Flower, Larkspur, Cranberry, Whortleberry, Purple Grape, Cranberry Sloe, Cherry Plum, Radish Plum and Red Beet are described by Willstätter and coworkers[20].

Evaporate ether solutions to dryness. warm the residue with a little alcohol and dilute the alcholic solution with water. Apply the reagents as stated below.

Hydrochloric acid.—Add concentrated acid (sp. gr. 1.20) to the solution, first 1 or 2 drops, then a large excess, equal to 3–4 times the volume of the solution.

Sodium hydroxid (*potassium hydroxid*).—Make the solution slightly alkaline by adding a drop of the 10 per cent sodium hydroxid solution. A 10 per cent solution of potassium hydroxid in methyl alcohol *must be used* for the "brown phase reaction" for chlorophyll, described under **23**, and may also be employed for the other tests.

Sodium hydrosulphite.—Add the sodium hydrosulphite solution drop by drop.

Ferric chlorid.—Add a small amount of the 0.5 per cent ferric chlorid solution to the solution to be tested. The reagent must be added very carefully, a small drop at a time, as the colorations are not obtained in all cases when an excess is used.

Alum.—Add to the test solution one-fifth its volume of the 10 per cent potassium or ammonium alum solution.

Uranium acetate.—Add the 5 per cent uranium acetate solution drop by drop to the solution to be tested.

Concentrated sulphuric acid on the dry color.—Evaporate a small amount of the solution or of the coloring matter in a porcelain dish. Cool thoroughly and treat the dry residue with 1 or 2 drops of cold, concentrated sulphuric acid. The colorations are in some cases extremely fugitive and may be observed only the instant the acid wets the residue.

TABLE 10.

Behavior of certain natural coloring matters with common reagents.

COLORING MATTER	HYDROCHLORIC ACID	SODIUM HYDROXID SOLUTION	SODIUM HYDROSULPHITE SOLUTION	FERRIC CLORID SOLUTION	ALUM SOLUTION	URANIUM ACETATE SOLUTION	CONCENTRATED SULPHURIC ACID ON DRY COLOR
Logwood	Deep red with excess of acid	Violet to violet-blue	Almost decolorized, color returning imperfectly by reoxidation	Dark shades of violet, brown or black (the first hue often evanescent)	Rose-red (change rather slow)	Violet, quickly fading	Red, changing to yellow
Red woods (Brazilwood, Sandalwood, Camwood and Barwood)	Deep red with excess of acid	Violet-red	....	Dark shades of violet, brown or black (the first hue often evanescent)	Rose-red (change rather slow)	....	....
Anthocyans of red fruit colors	....	Change to green, dull blue or slate color, usually very quickly, becoming browner by oxidation	Anthocyanidins derived by hydrolysis, almost completely decolorized	....	....	....	....
Alkanet	....	Deep blue	....	....	....	Yellowish green	Violet-blue
Archil	Little or no change	Blue	Decolorized, color returning when shaken with air. Reaction more easily seen in alkaline solution	....	....	....	Violet-blue

Cochineal	Little or no change	Violet	No marked change	Slightly darker	....	Green	....
Annatto	Remains orange. Little change	....	Little affected	No marked change. Perhaps somewhat browner	....	....	Blue
Turmeric (solution in ether or alcohol characterized by pure yellow color and light green fluorescence)	Orange-red or carmine-red on addition of several volumes of concentrated acid	Orange-brown	Little affected	No marked change. Perhaps somewhat browner	Little change	Somewhat browner	Red
Flavone colors of Fustic, Persian Berries, Quercitron, etc.	Becomes intensely yellow with 2–4 volumes of concentrated acid.	Bright yellow	Little affected	Olive-green or black colorations	More strongly yellow; Fustic, developing a green fluorescence	Orange colorations	Yellow to orange
Saffron	Little or no change	Remains yellow	Little affected	No marked change. Perhaps somewhat browner	Little change	Not affected	Blue
Carotin and Xanthopyll	Little change. Perhaps slightly paler	Little or no change	Little affected	....	....	....	Blue, reaction obtained with difficulty
Green Chlorophyll	More brownish	"Brown phase reaction", 23	....	....	....	....	....
Caramel	Little or no change	Little change or slightly deeper brown	Slightly paler	No change	....	....	....

SPECIAL TESTS FOR NATURAL COLORING MATTERS.

23 CHLOROPHYLL.

The "brown phase reaction"[21] may be useful for the characterization of chlorophyll, when this has not been previously treated with alkalies. Treat the green ether or petroleum ether solution of the coloring matter with a small amount of 10 per cent solution of potassium hydroxid in methyl alcohol. The color becomes brown, quickly returning to green.

24 ANNATTO[22].

Pour on a moistened filter an alkaline solution of the color obtained by shaking out the oil or melted and filtered fat with warm, dilute sodium hydroxid solution. If Annatto is present, the filter paper will absorb the color so that, when washed with a gentle stream of water, it will remain dyed a straw color. Dry the filter and add a drop of stannous chlorid solution. If the color turns pink the presence of Annatto is confirmed.

25 TURMERIC.

Carry out the highly characteristic reaction of Curcumine (Turmeric) with boric acid as follows: Treat the aqueous or dilute alcoholic solution of the color with hydrochloric acid until the shade just begins to appear slightly orange. Divide the mixture into two parts and add some boric acid powder or crystals to one portion. A marked reddening will be quickly apparent, best seen by comparison with the portion to which the boric acid has not been added. The test may also be made by dipping a piece of filter paper in the alcoholic solution of the coloring matter, drying at 100°C., then moistening with a weak solution of boric acid to which a few drops of hydrochloric acid have been added. On drying again a cherry-red color will be developed.

26 COCHINEAL.

When the presence of Cochineal is suspected, acidify the mixture with one-third its volume of concentrated hydrochloric acid and shake with amyl alcohol. Wash the amyl alcohol solution of the coloring matter 2–4 times with equal volumes of water to remove hydrochloric acid, etc. Dilute the amyl alcohol with 1–2 volumes of gasoline and shake with a few small portions of water to remove the color. Separate the solution into 2 portions. To the first add, drop by drop, 5 per cent uranium acetate solution, shaking thoroughly after each addition. In the presence of Cochineal a characteristic emerald-green color is produced[23]. The green coloration with uranium salts is not developed in the presence of much free acid. Therefore add a little sodium acetate before making this test or a correspondingly large amount of uranium acetate must be added. To the second portion add a drop or so of ammonium hydroxid, and, in the presence of Cochineal, a violet coloration results. This, however, is not so characteristic as the first test and many fruit colors give tests hardly to be distinguished.

As Cochineal lakes very often contain tin, further examination for this metal should always be made when water-insoluble Cochineal compounds appear to be present.

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XI. METALS IN FOODS.

ARSENIC[1].—TENTATIVE.

1 REAGENTS.

(**a**) *Nitric and sulphuric acids, arsenic-free.*—Specific gravities 1.42 and 1.84 respectively.

(**b**) *Sulphuric acid, arsenic-free* (*1 to* 2).

(**c**) *Zinc, arsenic-free.*—Stick zinc broken into pieces approximately 1 cm. in length.

(**d**) *Lead acetate paper.*—Heavy filter paper soaked in 20 per cent lead acetate solution, dried and cut into pieces about 4.5 by 16 cm.

(**e**) *Lead acetate cotton.*—Absorbent cotton soaked in 5 per cent lead acetate solution.

(**f**) *Mercuric bromid paper.*—Cut heavy, close-textured drafting paper (similar to Whatman's cold pressed) into strips exactly 2.5 mm. wide and about 12 cm. long. Soak for an hour in a 5 per cent solution of mercuric bromid in 95 per cent alcohol, squeeze out the excess of solution and dry on glass rods. Cut off the ends of the strips before using.

(**g**) *Potassium iodid solution.*—Containing 20 grams of potassium iodid per 100 cc.

(**h**) *Stannous chlorid solution.*—Forty grams of stannous chlorid crystals made up to 100 cc. with concentrated hydrochloric acid.

(**i**) *Standard arsenic solution.*—Dissolve 1 gram of arsenious oxid in 25 cc. of 20 per cent sodium hydroxid solution, neutralize with dilute sulphuric acid, add 10 cc. of the concentrated sulphuric acid and dilute to 1 liter with recently boiled water. One cc. of this solution contains 1 mg. of arsenious oxid (As_2O_3).

Dilute 20 cc. of this solution to 1 liter. Fifty cc. of the latter solution when diluted to 1 liter give a dilute standard solution containing 0.001 mg. of arsenious oxid (As_2O_3) per cc. which is used to prepare the standard stains. The dilute solutions must be prepared immediately before use.

2 APPARATUS.

Use a 2 ounce wide-mouthed bottle as a generator. Fit this by means of a perforated rubber stopper with a glass tube, 1 cm. in diameter and 6 cm. long, containing a piece of the lead acetate paper rolled into a cylinder. Connect this tube by means of a perforated rubber stopper with a similar tube filled with the lead acetate cotton, squeezed to remove excess of the solution. The cotton in all tubes used should be uniformly moist to obtain comparative stains. Connect the second tube by means of a perforated rubber stopper with a narrow glass tube, 3 mm. in internal diameter and 12 cm. long, containing a strip of the mercuric bromid paper. (See Fig. 5.) Rubber stoppers used for connections must be free from any white coating.

3 PREPARATION OF SOLUTION.

Weigh 5–50 grams of the finely divided and well-mixed sample into a porcelain casserole, the amount selected depending upon the character of the material and the ease with which it is oxidized. With dry, highly nitrogenous substances employ 5 grams; pulped vegetables, 25 grams, liquids with low solid content like beer or vinegar, 50 grams. Add 10–15 cc. of the nitric acid, cover the casserole by setting a watch glass inside the rim, convex side upward, heat until vigorous action is over, cool and add 10 cc. of the concentrated sulphuric acid. Heat on a wire gauze over a flame until the mixture turns dark brown or black, then add more nitric acid in 5 cc. portions, heating after each addition until the liquid remains colorless or yellow when evaporated until sulphur trixod fumes are evolved. To remove completely all nitric or nitrous acid, evaporate to about 5 cc., cool, dilute with 10–15 cc. of water and again evaporate until white fumes are evolved. Cool, dilute with water, again cool and make up with water to a definite volume (usually 25–100 cc., depending upon the amount of sample taken and its arsenic content).

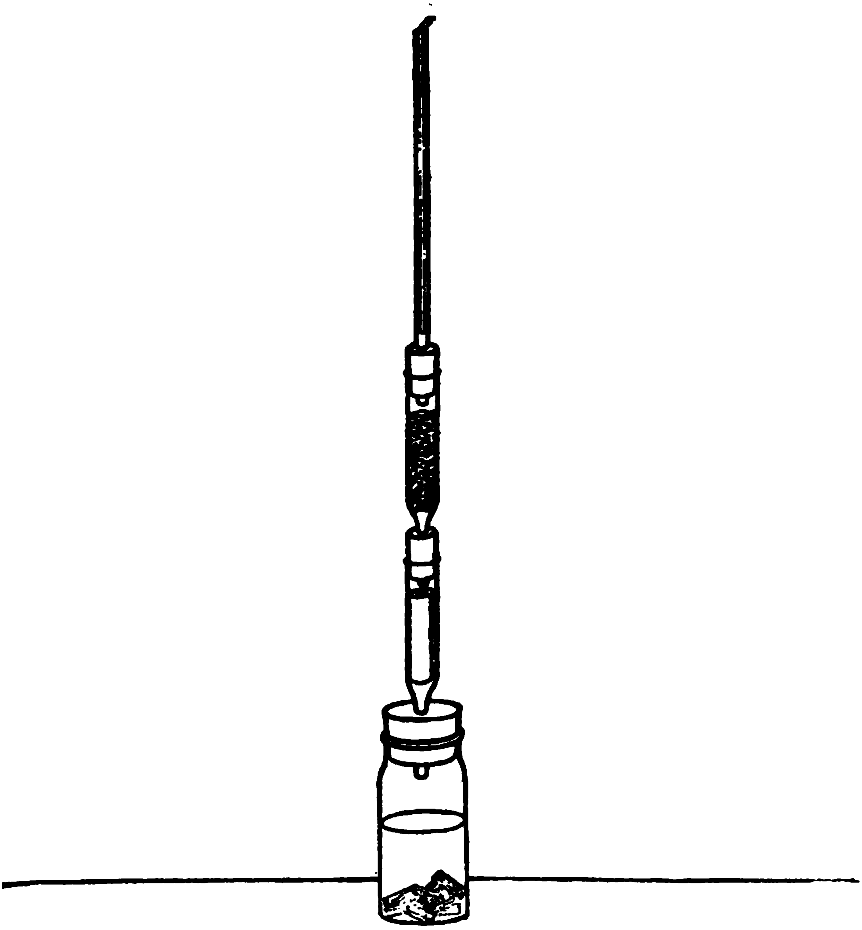

FIG. 5. APPARATUS FOR THE DETERMINATION OF ARSENIC.

4 DETERMINATION.

Introduce 20 cc. of the solution (or, if the amount of arsenic is large, an aliquot containing not more than 0.03 mg. of As_2O_3), prepared as directed under **3**, into the generator of the apparatus described in **2** and add 20 cc. of the dilute sulphuric acid. If the total volume is less than 40 cc., dilute to that volume with water and add 4 cc. of the 20 per cent potassium iodid solution. Heat to about 90°C., add 3 drops of the stannous chlorid solution and heat for 10 minutes. Cool the generator and its contents in a pan containing water and ice; when cold add about 15 grams of the stick zinc and connect the entire apparatus as described in **2**. Keep the bottles in ice water for 15 minutes, then remove from the bath and allow the evolution of gas to proceed for an hour longer. Remove the sensitized paper and compare the stain with similar ones produced under like conditions with known amounts of arsenic, using portions of the standard arsenic solution, containing 0.001, 0.002, 0.005, 0.010, 0.015, 0.025 and 0.030 mg. of arsenious oxid (As_2O_3), and adding such quantities of water and sulphuric acid that the same volume and acid strength are maintained as above.

Conduct a blank test on the reagents alone and correct the result for any arsenic so found. The blank should not exceed 0.001 mg.

TIN[2].

5 *Gravimetric Method.-Tentative.*

Weigh 50–100 grams of the sample (depending upon the amount of dry substance present and the relative ease with which the organic matter is oxidized) into an 800 cc. Kjeldahl flask and add 100 cc. of concentrated nitric acid. Allow to stand overnight (this procedure being preferred if much fat or sugar is present) or else place the flask on a wire gauze over a free flame and heat until the contents boil quietly. Add 25–50 cc. of concentrated sulphuric acid (depending upon the amount of dry substance present in the sample), and heat until white fumes are generated, cool somewhat, then add 5–10 cc. of concentrated nitric acid and continue heating as before. Repeat the addition of nitric acid until the solution remains clear after boiling off the nitric acid and fumes of sulphur trioxid appear.

Add 200 cc. of water to the digested sample, prepared as directed above, and pour into a 600 cc. beaker. Rinse out the Kjeldahl flask with three portions of boiling water so that the total volume of the solution is about 400 cc. Cool, add concentrated ammonia until just alkaline and then hydrochloric or sulphuric acid until the acidity is about 2 per cent. Place the beaker, covered, on a hot plate, heat to about 95°C. and pass in a slow stream of hydrogen sulphid for another hour. Digest on the hot plate for an hour and allow to stand 1–2 hours longer.

Filter the tin sulphid on an 11 cm. filter, similar in quality to No. 590, white ribbon, S. & S. Wash alternately with three portions each of wash solution (100 cc of saturated ammonium acetate solution, 50 cc. of glacial acetic acid and 850 cc. of water) and hot water. Digest the filter and precipitate in a 50 cc. beaker with three successive portions of ammonium polysulphid, heat to boiling each time and filter through a 9 cm. filter. Wash the precipitate on the filter with hot water. Acidify the filtrate with acetic acid, digest on a hot plate for an hour, allow to stand overnight and filter through a double 11 cm. filter. Wash alternately with two portions each of the wash solution and hot water and dry thoroughly in a weighed porcelain crucible. Ignite over a Bunsen flame, very gently at first and later at full heat. The crucible, partly covered, is then heated strongly with a large Bunsen or Meker burner. Stannic sulphid must be roasted gently to the oxid, which may be heated strongly without loss by volatilization. Weigh as stannic oxid and calculate to metallic tin, using the factor 0.7881.

Volumetric Method[8].—*Tentative.*

6 REAGENTS.

(a) *Air-free wash solution.*—Dissolve 20 grams of sodium bicarbonate in 2 liters of boiled water and add 40 cc. of concentrated hydrochloric acid. This solution should be freshly prepared.

(b) *N/100 iodin.*—The solution must be standardized frequently against (d), containing asbestos, and treated as described in 7, omitting the precipitation and boiling with hydrochloric acid and potassium chlorate. To obtain exact results the tin solution used for standardization should contain about the same amount of tin as is found in the sample under examination.

(c) *N/100 sodium thiosulphate.*

(d) *Standard tin solution.*—Dissolve 1 gram of tin in about 500 cc. of concentrated hydrochloric acid. Make up to 1 liter with water. One cc. contains 1 mg. of tin.

(e) *Sheet aluminium.*—Use sheet aluminium, about 30 gauge, free from tin.

7 DETERMINATION.

Proceed as directed under 5 to "Digest on the hot plate for an hour and allow to stand 1–2 hours longer."

Filter the precipitate of tin sulphid upon asbestos in a Gooch crucible with a detachable bottom, using suction. Wash the precipitate a few times and then transfer the detachable bottom, asbestos pad, and tin precipitate to a 300 cc. Erlenmeyer flask. Remove all traces of the precipitate from the inside of the crucible by means of a jet of hot water and a policeman, using a minimum amount of water for washing.

Add to the flask 100 cc. of concentrated hydrochloric acid and 0.5 gram of potassium chlorate. Boil for about 15 minutes, making about 4 more additions of smaller amounts of potassium chlorate as chlorin is boiled out of the solution. Wash the particles of potassium chlorate down from the neck of the flask with water and finally boil to remove chlorin. Then add about 1 gram of the sheet aluminium to dispel the last traces of chlorin.

Attach the flasks, in duplicate, as described below, to a large carbon dioxid generator. Pass the carbon dioxid through a scrubber containing water and then divide into two streams by means of a Y-tube, each stream of carbon dioxid entering one of the flasks by means of a long rubber tube connected with a bulbed tube, passed through the rubber stopper of the flask and having its lower end near the surface of the liquid in the flask. The carbon dixoid leaves the flask by a second bulbed tube, the opening of which is near the top of the flask. This glass tube is connected by a long rubber tube to a second glass tube about 10 inches long which is immersed in a cylinder containing water. This gives a water-seal to the delivery tube and a pressure against which the current of carbon dioxid must work. It also restrains any strong flow of gas when not desired and permits a gas pressure in the Erlenmeyer flask.

After the flasks are connected, raise the tubes in the water-seal cylinders so that the generator has practically no pressure to overcome. Allow the carbon dioxid to run for a few minutes. Drop the tubes to the bottom of the cylinders, creating pressure in the flasks. Lift the rubber stoppers of the flasks alternately about a dozen times, in order to force out any air remaining in the flasks. Slightly raise the stopper on one of the flasks and quickly drop about 2 grams of sheet aluminium into the flask. The aluminium should be folded into a strip about 1 cm. wide and slightly bent so as to prevent it striking directly on the bottom of the flask. After the aluminium has entirely dissolved, raise the tubes in the water-seal cylinders so as to allow carbon dioxid to pass through, place the flasks upon hot plates, and heat to boiling. After boiling for a few minutes, remove the flasks from the hot plates and cool in ice water (or cold running water), still maintaining within them an atmosphere of carbon dioxid. Lower the tubes in the cylinder. When cool, disconnect the flasks one at a time, putting a glass plug into the tube through which the carbon dioxid enters. Wash the tubes, rubber stopper and sides of the flask with the air-free wash solution, add starch paste and titrate at once with the N/100 iodin.

If it is desired to titrate by the excess method, run an excess of the N/100 iodin into the flask while it is still connected with the carbon dioxid stream. Then wash out the tubes and titrate the excess of iodin with the N/100 sodium thiosulphate.

The rubber connections should be washed with water after each determination.

8 COPPER.—TENTATIVE.

Destroy organic matter as directed under **5**. Concentrate the sulphuric acid residue, by continued digestion, to a volume of 10–15 cc., cool, dilute with a little water, transfer to a 400 cc. beaker, rinse the Kjeldahl flask with water, adding the rinsings to the contents of the beaker, dilute to about 200 cc. and boil to expel nitrous fumes. Cool, render the solution slightly alkaline with ammonium hydroxid and boil to expel the excess of ammonia. Add 5 cc. of concentrated hydrochloric acid for each 100 cc. of solution, heat to incipient boiling and saturate the solution with hydrogen sulphid. Allow to stand on a steam bath for a few minutes until the sulphid flocculates, filter and wash the precipitate with hydrogen sulphid water. Protect the precipitate from contact with air as much as possible, use only hydrogen sulphid water for washing and carry out this operation without interruption. Reserve the filtrate for the determination of zinc, if necessary. Place the filter containing the copper sulphid precipitate in a small flask, add 4–5 cc. of concentrated sulphuric acid and the same amount of nitric acid and heat until white fumes appear. Continue the oxidation, adding a little nitric acid from time to time, until the liquid remains colorless upon heating to the appearance of white fumes. Cool, dilute with about 30 cc. of water, add an excess of bromin water and boil until all bromin is expelled. Determine the copper as directed under **VII, 28**, using N/100 sodium thiosulphate for the titration.

9 ZINC.—TENTATIVE.

Proceed as directed under **8** to the point indicated by the sentence "Reserve the filtrate for the determination of zinc, if necessary". Boil the filtrate, containing the zinc, to expel hydrogen sulphid and to reduce the volume to about 250–300 cc., add a drop of methyl orange and 5 grams of ammonium chlorid and make alkaline with ammonium hydroxid. Add dilute hydrochloric acid, drop by drop, until the reaction is faintly acid, then add 10–15 cc. of 50 per cent sodium or ammonium acetate solution and pass in hydrogen sulphid for a few minutes until precipitation is complete. Allow the precipitate to settle, filter, refilter, if necessary, until the filtrate is clear and wash the precipitate twice with hydrogen sulphid water. Dissolve the precipitate on the filter with a little hydrochloric acid (1 to 3), wash the filter with water, boil the filtrate and washings to expel hydrogen sulphid, cool and add a distinct excess of bromin water. Then add 5 grams of ammonium chlorid and ammonium hydroxid until the color, caused by free bromin, disappears. Add hydrochloric acid (1 to 3), drop by drop, until the bromin color just reappears, then add 10–15 cc. of sodium or ammonium acetate solution (50 per cent by weight) and 0.5 cc. of ferric chlorid solution (10 grams per 100 cc.), or enough to precipitate all the phosphates. Boil until all the iron is precipitated. Filter while hot and wash the precipitate with water containing a little sodium acetate. Pass hydrogen sulphid into the combined filtrate and washings until all the zinc sulphid, which should be pure white, is precipitated, filter upon a tared Gooch crucible and wash with hydrogen sulphid water, containing a little ammonium nitrate. Dry the crucible and its contents in an oven, ignite at a bright red heat, cool and weigh as zinc oxid. Calculate the weight of metallic zinc, using the factor 0.8034.

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XII. FRUITS AND FRUIT PRODUCTS.

1 PREPARATION OF SAMPLE.—OFFICIAL.

All samples received in open packages (i. e., not in sterile condition) must be transferred without delay to glass-stoppered containers and kept in a cool place. The determination of alcohol, total and volatile acids, solids and sugars, particularly in the case of fruit juices and fresh fruits, should be made at once, as fermentation is liable to begin very soon. Portions for the determination of sucrose and reducing sugar may be weighed and, after adding a slight excess of neutral lead acetate solution, kept without fermenting for several days if desired. The various products are prepared as directed below:

(a) *Juices.*—Mix thoroughly by shaking to insure uniformity in sampling. Remove any extraneous matter by decantation or by filtering through muslin. Fresh juices may be prepared by pressing the well-pulped fruit in a jelly bag and filtering through muslin. In case of citrus fruit express the juice by means of one of the common devices for squeezing oranges or lemons, using the entire fruit for this purpose, and strain the expressed juice through muslin.

(b) *Jellies and sirups.*—Mix thoroughly to insure uniformity in sampling. Weigh 60 grams into a 300 cc. flask, add water, dissolve by frequent shaking, then make up to the mark with water and use aliquots for the various determinations. If the jelly contains starch or other insoluble material, mix thoroughly before taking the aliquots.

(c) *Fresh and dried fruits.*—Pulp the whole, well-cleaned fruit in a large mortar or by means of a food chopper and mix thoroughly. In the case of stone fruits, remove the pits and determine their proportion in a weighed sample.

(d) *Jams, marmalades, preserves and canned fruits.*—Pulp thoroughly the entire contents of the jar or can, as directed under (c); remove the pits from stone fruits and, if desired, determine their proportion in a weighed sample. In the examination of canned fruits it is often sufficient merely to examine the sirups in which the fruits are preserved. In such cases the liquor may be separated and treated as prescribed for juices.

2 ALCOHOL.—OFFICIAL.

Determine alcohol in 50 grams of the original material as directed under VIII, 28.

3 TOTAL SOLIDS.—OFFICIAL.

(a) *Juices, jellies and sirups containing no insoluble matter.*—Proceed as directed under VIII, 3, 5, 7 or 9, employing the sample prepared as directed in 1 (a) or (b).

(b) *Fresh and dried fruits, jams, marmalades, preserves, canned foods and other products containing insoluble matter.*—Weigh about 20 grams of pulped fresh fruit, or such an amount of fruit products as will give not more than 3–4 grams of dried material; if necessary to secure a thin layer of the material, add a few cc. of water, mix thoroughly and dry as directed under VIII, 3 or 4.

It is to be noted that certain State and Federal regulations require the moisture in dried apples to be determined by drying for 4 hours at the temperature of boiling water.

INSOLUBLE SOLIDS.

4 *Direct Method.—Official.*

Transfer 50 grams of the sample to a mortar by means of warm water and macerate thoroughly; then transfer to a muslin filter and wash thoroughly with about 500 cc. of warm water, stirring the pulp thoroughly on each addition of water. This amount of water is usually sufficient to remove all soluble material. In extreme cases increase the washings to 1 liter. Transfer the insoluble residue to an evaporating dish, dry and weigh. If it is desired to determine the alcohol precipitate, **18**, cool the filtrate, make up to a definite volume and reserve for this determination.

5 *Indirect Method.—Official.*

Transfer 25 grams of the fruit product to a 250–500 cc. graduated flask, the size of the flask depending upon the volume of insoluble matter present, add water, shake thoroughly and make up to volume. Allow to settle and either filter or decant the supernatant liquid. Determine the soluble solids in an aliquot, as directed under **3 (a)**. The fruit must be macerated thoroughly; the use of a mechanical shaker is advisable. The percentage of insoluble solids is the difference between the percentage of the total solids and the percentage of soluble solids.

6 TOTAL ASH.—OFFICIAL.

Determine the ash as directed under **VII, 4**, using 50 cc. of the solution of the jelly or diluted sirup, **1 (b)**, evaporated to dryness, or 25 grams of juice or of fresh or canned fruit, or 10 grams of jam, marmalade, preserves or dried fruit.

7 ALKALINITY OF THE ASH.—OFFICIAL.

Into the platinum dish containing the ash introduce a measured excess of N/5 nitric acid, heat to boiling, cool and add a few drops of methyl orange. Carefully rub up the ash with a rubber-tipped stirring rod and titrate the excess of acid with N/10 potassium or sodium hydroxid. Express the result as the number of cc. of N/10 acid required to neutralize the ash from 100 grams of the sample.

8 SULPHATES AND CHLORIDS.—OFFICIAL.

Wash the solution of the ash, obtained in **7**, into a 50 cc. flask and make up to the mark with water. Evaporate 25 cc. of this solution to dryness several times with concentrated hydrochloric acid, take up the final residue in a small amount of hot water, filter, wash the paper with hot water, acidify the filtrate with a few drops of hydrochloric acid and determine the sulphates by precipitation with barium chlorid solution. From the weight of barium sulphate calculate the sulphates present as per cent of potassium sulphate, using the factor 0.7465.

In the other portion of the solution determine the chlorids as directed under **II, 17**. The nitric acid added before making the titration will, if it contain enough nitrous oxid, completely destroy the red color of the methyl orange and leave a clear solution for the titration. Calculate the chlorids as per cent of sodium chlorid.

9 TOTAL ACIDITY.—OFFICIAL.

Dilute 25 cc. of the solution of jelly or diluted sirup, **1 (b)**, or 10 grams of juice or fresh fruit with recently boiled water to about 250 cc., or less if the sample be not highly colored; titrate the acid with N/10 alkali, using phenolphthalein as an indicator.

In the case of highly colored products employ azolitmin solution or phenolphthalein powder [**XV**, (**23**)] on a spot plate instead of phenolphthalein solution. Calculate the results as malic, citric or tartaric acid, specifying the acid used and expressing the results in per cent or grams per 100 cc.

10 VOLATILE ACIDS.—OFFICIAL.

Dissolve 10 grams of the sample, dilute to 25 cc. and distil in a current of steam, as directed under **XV**, **25**. Each cc. of N/10 alkali is equivalent to 0.0060 gram of acetic acid.

11 FREE MINERAL ACIDS.—TENTATIVE.

Proceed as directed under **XVIII**, **26**, **27** or **28**.

12 PROTEIN.—OFFICIAL.

Proceed as directed under **I**, **18**, **21** or **23**, using 5 grams of jelly or other fruit product containing a large amount of sugar, or 10 grams of juice or fresh fruit and a larger quantity of the sulphuric acid if necessary for complete digestion. Multiply the percentage of nitrogen by 6.25 to obtain the percentage of protein.

SUCROSE.

13 *By Polarization—Official.*

Determine by polarizing before and after inversion, as directed under **VIII**, **19** or **20**.

14 *By Reducing Sugars Before and After Inversion.—Official.*

Proceed as directed under **VII**, **18**.

15 REDUCING SUGARS.—OFFICIAL.

Proceed as directed under **VII**, **25**, expressing the results as invert sugar.

16 COMMERCIAL GLUCOSE.—OFFICIAL.

Proceed as directed under **VIII**, **22**.

17 DEXTRIN.—TENTATIVE.

Dissolve 10 grams of the sample in a 100 cc. flask, add 20 mg. of potassium fluorid, and then about one-fourth of a cake of compressed yeast. Allow the fermentation to proceed below 25°C. for 2–3 hours to prevent excessive foaming, and then incubate at 27°–30°C for 5 days. At the end of that time, clarify with basic lead acetate solution and alumina cream, make up to 100 cc. and polarize in a 200 mm. tube. A pure fruit jelly will show a dextro or laevo rotation of not more than a few tenths of a degree. If a polariscope having the Ventzke scale be used and a 10 per cent solution polarized in a 200 mm. tube, the number of degrees read on the sugar scale of the instrument, multiplied by 0.8755, will give the percentage of dextrin, or the following formula may be used:

$$\text{Percentage of dextrin} = \frac{C \times 100}{198 \times L \times W} \text{ in which}$$

C = degrees of circular rotation;
L = length of tube in decimeters;
W = weight of sample in 1 cc.

18

ALCOHOL PRECIPITATE.—OFFICIAL.

Evaporate 100 cc. of a 20 per cent solution of jelly or diluted sirup, **1 (b)**, or of the washings from the determination of insoluble solids, **4**, to 20 cc.; add slowly, with constant stirring, 200 cc. of 95 per cent alcohol by volume and allow the mixture to stand overnight. Filter and wash with 80 per cent alcohol by volume. Wash the precipitate from the filter paper with hot water into a platinum dish; evaporate to dryness; dry at 100°C. for several hours and weigh; then burn off the organic matter and weigh the residue as ash. Designate the loss in weight upon ignition as the alcohol precipitate.

The ash should be chiefly lime and not more than 5 per cent of the total weight of the alcohol precipitate. If it is greater than this, some of the salts of the organic acids have been brought down. Titrate the water-soluble portion of this ash with N/10 acid, as any potassium bitartrate precipitated by the alcohol can thus be estimated.

STARCH.

19

Qualitative Test.—Official.

Dilute a portion of the sample with water, heat nearly to boiling, add several cc. of dilute sulphuric acid and then add potassium permanganate solution until all color is destroyed. Cool and test with iodin solution. The presence of starch is not necessarily an indication of its addition as an adulterant. It is usually present in small amount in the apple, and occasionally in other fruits, and unless it is found in the fruit product in considerable amount its presence may be due to these natural sources.

GELATIN[1].

20

Qualitative Test.—Tentative.

The presence of gelatin in jellies and jams is shown by the increased content of nitrogen. Precipitate a concentrated solution of jelly or jam with 10 volumes of absolute alcohol and determine nitrogen in the dried precipitate as directed under **I**, **18**, **21** or **23**.

AGAR AGAR.

21

Detection by Microscopic Examination.[2]*—Tentative.*

Heat the jelly with 5 per cent sulphuric acid, add a crystal of potassium permanganate and allow to settle. If agar agar is present the sediment will be rich in diatoms, which can be detected by the use of the microscope.

22

Detection by Precipitation[3]*.—Tentative.*

Cover 30 grams of the jam or jelly with 270 cc. of hot water, stir until thoroughly disintegrated and boil for 3 minutes. Filter immediately, while still boiling hot, through a filter paper of texture similar to No. 597, S. & S. In the presence of agar agar a precipitate will form upon standing not longer than 24 hours. Filter, wash with cold water and dissolve from the paper by means of a very small amount of boiling water. Upon chilling this hot water solution a firm jelly will be formed that can be examined by the touch. This method will detect 0.2 per cent of agar agar with certainty if the proportions of jam or jelly and water are strictly observed.

TARTARIC ACID.—TENTATIVE.

23

PREPARATION OF SOLUTION.

Filter fruit juices and employ the filtrate directly. In the case of jellies filter the solution, prepared as directed under **1 (b)**, and employ the filtrate. In the case of sirups or substances containing insoluble matter like pulped fruit, jams, marmalades, etc., weigh 50-100 grams, the amount selected being dependent upon the content of solids, of the sample, prepared as directed in **1** (**c**) or (**d**). Introduce into a 200 cc. graduated flask, make up to the mark with water, allow to stand for an hour, shake at frequent intervals, filter through a dry paper and use the filtrate.

24 DETERMINATION.

Determine the tartaric acid in 100 cc. of fruit juice or the same amount of a solution of the sample, prepared as directed under **23**, employing the method given under **XV**, **27**, except that 20 cc. of alcohol are used in the precipitation instead of 15 cc.

MALIC ACID.

25 *Method I*[4].—*Tentative.*

(For fruit juices and similar products containing no tartaric acid and not over 15 per cent of sugars and in which the color does not interfere with polarization.)

Filter the sample, if necessary to secure a solution which can be readily polarized, and polarize with white light, using a 200 mm. tube if possible.

If the sample contains free mineral acid, transfer a measured portion (75 cc. is a convenient volume) to a 100 cc. graduated flask, add enough standard alkali, calculate from the acidity as determined in **9**, to neutralize the total acidity, dilute to the mark, mix well and filter. If no free mineral acids are present, it is unnecessary to neutralize the sample. If neutralized, proper correction must be made for dilution in making the final calculation.

Transfer 25 cc. of the sample, or of the neutralized solution, to a flask graduated at 25 and 27.5 cc., add about 2.5 grams of powdered uranyl acetate and shake vigorously at frequent intervals for 3 hours, keeping the mixture well protected from light. If all of the uranyl acetate dissolves, add more so that a small amount remains undissolved at the end of 3 hours. Dilute the solution to the 27.5 cc. mark with saturated uranyl acetate solution, mix well and filter, if necessary, through a folded filter. Polarize, if possible, in a 200 mm. tube. If the solution is too dark to polarize in a 200 mm. tube, a 100 or 50 mm. tube may be used. Multiply the reading by 1.1 to correct for the dilution.

Multiply the algebraic difference in degrees Ventzke between the two readings calculated to the basis of a 200 mm. tube by the factor 0.036 to obtain the weight of malic acid in the sample in grams per 100 cc.

Make all polarizations at the same room temperature with white light. Make at least six readings in each case and take an average of these.

In the case of dark colored fruit juices which can not be polarized readily, approximately quantitative results may be obtained by adding to the solutions a few drops of bromin, shaking thoroughly and filtering just before polarization.

Method II.—*Tentative.*

(Approximate determination for fruit juices and similar products containing no tartaric acid and more than 15 per cent of sugars.)

26 PREPARATION OF SOLUTION[5].

Weigh out 25 grams of the sample and transfer to a 600 cc. beaker with a little 95 per cent alcohol by volume. Add alcohol a little at a time until 200 cc. have been added, stirring the mixture well, and warming, if necessary, to insure solution of all alcohol-soluble substances. Filter on a Büchner funnel, using suction, and thoroughly wash the precipitated pectins and insoluble matter with 95 per cent alcohol, disregarding any slight turbidity which may appear in the filtrate after the washings have been added. From **9**, calculate the amount of N/4 barium hydroxid required nearly to neutralize the acidity in the 25 grams of sample taken. To the combined filtrate and washings in an Erlenmeyer flask add the calculated quantity of barium hydroxid solution, stir until reaction is complete and then add 3-5 drops, or more if required, of 50 per cent barium acetate solution to insure an excess of barium. Make up the volume of the mixture to about 375 cc. (not less) with alcohol, and reflux until the precipitate settles readily after being shaken. This may require 3-4 hours. Filter with suction and thoroughly wash the precipitate in the flask and on the paper with 95 per cent alcohol by volume. Transfer the portion on the filter to the original flask, rinsing the paper with a jet of hot water. Digest the precipitate with hot water, containing 2 grams of sodium sulphate in solution, until the reaction is complete, and boil until the barium sulphate precipitate settles readily. Concentrate by evaporation, if necessary, and transfer to a 100 cc. volumetric flask with a little hot water, cool, make up to volume with water and filter.

27 DETERMINATION.

Transfer 25 cc. of the filtrate, obtained in **26**, to a flask graduated at 25 and 27.5 cc., add about 2.5 grams of pulverized uranyl acetate and shake vigorously at frequent intervals for 3 hours, keeping the solution well protected from light. If all the uranyl acetate dissolves, add more so that a small amount remains undissolved at the end of 3 hours. Dilute the solution to the 27.5 cc. mark with saturated uranyl acetate solution, mix well, filter if necessary, and polarize in a 200 mm. tube, using the same precautions as described in **25**. Multiply the reading, calculate to the basis of a 200 mm. tube, by 1.1 to correct for dilution.

Polarize another portion of the filtrate, obtained in **26**, which has not been treated with uranyl acetate. Multiply the algebraic difference in degrees Ventzke between the two readings, calculated to the basis of a 200 mm. tube, by the factor 0.036 to obtain the weight of malic acid in grams per 100 cc. in the solution as obtained in **26**.

Method III.—Tentative.

(Approximate determination for products containing tartaric acid).

28 PREPARATION OF SOLUTION[6].

Prepare the sample as directed under **26** up to the point of filtration and washing of the barium malate precipitate, then dry the precipitate thoroughly and transfer the portion on the filter to the original flask, rinsing the paper with a jet of hot water. Digest the precipitate with hot water, transfer to a 100 cc. volumetric flask with a little hot water, cool, make up to volume with water and filter to remove insoluble barium tartrate. This amount of water is sufficient to dissolve barium malate up to amounts as large as approximately 0.9 gram. More than 100 cc. of water must be used when more than 0.9 gram of barium malate is present. The amount of barium tartrate dissolved by hot water is so small as to affect only slightly the polarization after treatment with uranyl acetate.

29 DETERMINATION.

Proceed as directed under **27**, using the solution prepared as directed under **28**.

CITRIC ACID[7].—TENTATIVE.

(Applicable in the presence of sugar and malic and tartaric acids.)

30 REAGENTS.

(**a**) *Barium hydroxid solution.*—Approximately N/4.

(**b**) *Barium acetate solution.*—Dissolve 50 grams of barium acetate in water and dilute to 100 cc.

(**c**) *Sulphuric acid (1 to 1) and (1 to 5).*

(**d**) *Potassium or sodium bromid solution.*—Dissolve 15 grams of potassium bromid in 40 cc. of water or 16 grams of sodium bromid in 50 cc. of water.

(**e**) *Potassium permanganate solution.*—Dissolve 5 grams of potassium permanganate in water and dilute to 100 cc.

(**f**) *Ferrous sulphate solution.*—Dissolve 20 grams of ferrous sulphate in 100 cc. of water containing 1 cc. of concentrated sulphuric acid.

(**g**) *Bromin water.*—Freshly prepared, saturated solution.

31 DETERMINATION.

Proceed as directed under **26** up to "Filter with suction and thoroughly wash the precipitate in the flask and on the paper with 95 per cent alcohol by volume". Transfer the precipitate from the filter to the flask with a jet of hot water, boil until alcohol can no longer be detected by odor and add enough of the sulphuric acid (1 to 5) to precipitate all the barium originally added and to allow 2 cc. in excess. Evaporate by careful boiling to a volume of 60–70 cc., cool and add 5 cc. of freshly prepared saturated bromin water, or enough to show a distinct excess. Transfer with water to a 100 cc. volumetric flask and dilute to the mark at standard temperature. Mix thoroughly, allow the precipitate to settle and filter through a dry paper. The precipitate may be separated by centrifugalizing and the supernatant liquid decanted, if necessary. Pipette an aliquot of the filtrate, containing not more than 400 mg. of citric acid, calculated from the total acidity of the sample, into a 300 cc. Erlenmeyer flask. If possible, the amount of citric acid in the aliquot should exceed 50 mg. Add 10 cc. of the sulphuric acid (1 to 1) and 5 cc. of the potassium or sodium bromid solution, mix, warm the flask in a water bath to 48°–50°C. and allow it to remain in the bath for 5 minutes. After removing from the bath add rapidly from a burette 25 cc. of the potassium permanganate solution, drop by drop with frequent interruptions, and with constant, vigorous shaking, avoiding a temperature during oxidation exceeding 55°C. Set the flask aside until the hydrated peroxid of manganese begins to settle. The supernatant liquid should be dark brown, showing an excess of permanganate; if an excess is not indicated, add more permanganate. Shake again, set aside to settle and repeat this operation until the precipitate assumes a yellow color and most of it has dissolved. Finally, while the solution is still warm, remove the last undissolved portion of hydrated peroxid of manganese precipitate and also the excess of bromin by adding, drop by drop, the clear ferrous sulphate solution. Allow the solution to cool, shaking occasionally. If the operations have been properly conducted, a heavy white precipitate of pentabromacetone is obtained which becomes crystalline on occasional shaking and in this condition is entirely insoluble in water. Allow the mixture to stand overnight, collect it by means of gentle suction on a tared Gooch crucible provided with a thin pad of asbestos, previously dried over sulphuric acid in a vacuum desiccator, wash with water slightly acidified with sulphuric acid and finally wash twice with water. Dry the precipitate to constant weight over sulphuric acid in a vacuum desiccator, protecting the precipitate from strong light. The weight of pentabromacetone, multiplied by the factor 0.424, gives the equivalent weight of anhydrous citric acid ($H_3C_6H_5O_7$). Occasionally the pentabromacetone is first obtained in the form of oily droplets. These become crystalline on standing or on cooling and are usually discolored by negligible traces of manganese or iron.

The above method may be applied directly to the sample without previous precipitation of the citric acid as the barium salt when the amount of sugar or other permanganate reducing substances is not excessive. In this case begin the determination with the addition of 2 cc. of sulphuric acid (1 to 5) and the treatment with bromin water.

32 METALS.—TENTATIVE.

Proceed as directed under **XI**.

33 PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX**.

34 COLORING MATTERS.—TENTATIVE.

Proceed as directed under **X**.

35 SWEETENING SUBSTITUTES.

Proceed as directed under **IX**, **12**, **14**, **37** or **38**.

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XIII. CANNED VEGETABLES.

1 PHYSICAL EXAMINATION[1].—TENTATIVE.

Note carefully the external appearance of the packages to detect the presence of "leakers", "swells" or "springers". In general the ends of sound tins of canned vegetables are slightly concave. On opening the package note the relative proportion of solid and liquid contents and the level of the solids and of the total contents in the tin. Note the general appearance, odor, flavor, color and size of the vegetables; appearance of the liquor or brine, whether clear or turbid, and the condition of the inner walls of the container, especially as to blackening and corrosion. In all instances the analyst should familiarize himself with the normal appearance, odor, color, flavor and other properties of the product under examination. Careful macroscopic or microscopic examination should be made for worm infestation, mold, dirt or other evidence of decomposition or filth.

2 PREPARATION OF SAMPLE.—OFFICIAL.

The preparation of the sample for analysis depends upon the character of the product and the determinations to be made. Samples in which only the solid or liquid portion is required should be treated as follows: Weigh the full can, open, pour off the liquid, allow the solid portion to drain for a minute, re-weigh the can and drained vegetables, then remove the solid portion and weigh the dry, empty can. The method selected for draining the vegetables is dependent upon the nature and condition of the sample. In most cases it is sufficient to cut around the cover and before turning it back allow the liquor to drain through the slit. Whenever a portion of the solid material would escape with the liquor by this procedure, drain upon a piece of cheese-cloth. From the weights thus obtained determine the percentage of liquid and solid contents. If only the solid portion is required, separate in a similar manner and thoroughly grind the drained vegetables in a mortar or food chopper. If a composite of the solid and liquid portion is required, thoroughly grind the contents of the can in a mortar or food chopper. In all cases mix thoroughly the portion used and preserve the balance in glass-stoppered containers. Unless the analysis is to be completed in a reasonably short time, determine the moisture in a portion of the sample prepared as above and, in order to prevent decomposition, dry the remainder and then expose to air until it becomes air-dry, grind, mix thoroughly and preserve in glass-stoppered containers. A second moisture determination is required in this procedure.

3 MOISTURE.—OFFICIAL.

Dry a quantity of the sample, representing about 2 grams of dry material, as directed in VIII, 2.

4 ASH.—OFFICIAL.

Determine total ash as directed in VII, 4.

5 SALT.—OFFICIAL.

Determine chlorin as directed under II, 15 or 17, express the result in terms of sodium chlorid.

6

SUGARS.—TENTATIVE.

Determine reducing sugars and sucrose as directed in **VII, 57** and **58**, varying the weight of the sample employed according to its sugar content.

7

TOTAL ACIDS.—OFFICIAL.

Proceed as directed in **XV, 23**. Express the result as the number of cc. of N/10 alkali required to neutralize 100 grams of sample.

8

VOLATILE ACIDS.—OFFICIAL.

Proceed as directed in **XV, 25**. Express the results as acetic acid; 1 cc. of N/10 alkali is equivalent to 0.0060 gram of acetic acid.

9

PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX**.

10

COLORING MATTERS.—TENTATIVE.

Proceed as directed under **X**.

11

METALS.—TENTATIVE.

Proceed as directed under **XI**.

TOMATO PRODUCTS.

12

PREPARATION OF SAMPLE.—OFFICIAL.

Shake the package and contents thoroughly to incorporate any sediment, then transfer the entire contents of the container to a large glass or porcelain dish and mix thoroughly, continuing the stirring for at least 1 minute. Transfer the well-mixed sample to a glass-stoppered container and shake or stir thoroughly each time before removing portions for analysis.

13

TOTAL SOLIDS.—TENTATIVE.

Weigh 10 grams of the sample into a flat-bottomed dish having a diameter of about 6 cm., spread the sample in a thin layer, evaporate to dryness on a steam bath and dry in a water oven for 4 hours.

14

INSOLUBLE SOLIDS.—TENTATIVE.

Wash 20 grams of the sample repeatedly with hot water, centrifugalizing after each addition of water and pouring the clear, supernatant liquid through a tared triple filter paper on a Büchner funnel. After 4 or 5 washings transfer the remaining insoluble matter to the filter, dry for 2 hours at 100°C., cool in a desiccator and weigh rapidly. The paper used should have been dried previously for 2 hours at 100°C., cooled in a desiccator and weighed. A cylinder, 1–1¼ inches in diameter and 5–6 inches long, is convenient for washing and centrifugalizing.

15

SOLUBLE SOLIDS.—TENTATIVE.

Subtract the percentage of insoluble solids from the percentage of total solids to obtain the percentage of soluble solids.

16 SAND.—TENTATIVE.

Weigh 100 grams of the well-mixed sample into a 2-3 liter beaker, nearly fill the beaker with water, and mix the contents thoroughly. Allow to stand 5 minutes and decant the supernatant liquid into a second beaker. Refill the first with water and again mix the contents. After 5 minutes more decant the second beaker into a third, the first into the second, refill and again mix the first. Continue this operation, decanting from the third beaker into the sink until the lighter material is washed out from the sample. Then collect the sand from the three beakers on a tared Gooch crucible, dry, ignite and weigh. Attention is especially called to the fact that under "Sand" only the figure obtained by this method should be reported. The results obtained by the determination of ash insoluble in hydrochloric acid are not applicable to the determination of sand, since the sand is so unevenly distributed that reliable results can be obtained only by taking a larger sample than is possible in the determination of ash.

17 ASH.—OFFICIAL.

Evaporate 10 grams of the sample to dryness on a water bath and ignite as directed under **VII, 4**.

18 ALKALINITY OF THE ASH.—OFFICIAL.

Proceed as directed under **XII, 7**. Express the result as the number of cc. of N/10 acid required to neutralize the ash from 100 grams of the sample.

19 SODIUM CHLORID.—OFFICIAL.

Proceed as directed under **II, 15** or **17**, using either an aliquot of the solution obtained in **18** or a nitric acid solution of the whole ash.

20 REDUCING SUGARS BEFORE INVERSION.—OFFICIAL.

Weigh 20 grams of the sample into a 200 cc. flask, dilute with about 100 cc. of water, clarify with a slight excess of neutral lead acetate solution, dilute to the mark and filter. Remove the excess of lead with anhydrous sodium or potassium oxalate. Filter and determine reducing sugars as directed under **VII, 25**. Express the result as per cent of invert sugar.

21 REDUCING SUGARS AFTER INVERSION.—OFFICIAL.

Transfer 50 cc. of the filtrate, obtained in **20**, to a 100 cc. flask, add 5 cc. of concentrated hydrochloric acid and let stand overnight. Nearly neutralize with sodium hydroxid solution, cool, dilute to the mark and determine reducing sugars in an aliquot as directed under **VII, 25**. Express the result as per cent of invert sugar.

22 SUCROSE.—OFFICIAL.

Proceed as directed under **VII, 18**.

23 TOTAL ACIDS.—OFFICIAL.

Proceed as directed under **XVIII, 17**, employing 5 grams of the sample. Express the result as anhydrous citric acid. One cc. of N/10 alkali is equivalent to 0.0064 gram of anhydrous citric acid.

24 VOLATILE ACIDS.—OFFICIAL.

Proceed as directed under **XV, 25**, employing 25 grams of the sample, increasing the amount of water used for the distillation and collecting a correspondingly larger amount of distillate. Express the result as acetic acid. One cc. of N/10 alkali is equivalent to 0.0060 gram of acetic acid. Reserve the neutralized distillate for the detection of butyric acid.

BUTYRIC ACID.

25

Qualitative Test.—Tentative.

Evaporate the neutralized distillate, obtained in **24**, to dryness on a steam bath. Decompose the residue with about 5 cc. of 10 per cent sulphuric acid and note the odor.

26

FIXED ACIDS.—OFFICIAL.

Multiply the percentage of volatile acids, **24**, by 1.067 and subtract the product from the percentage of total acids, **23**, to obtain the per cent of fixed acids as citric acid.

MICRO-ANALYSIS OF TOMATO PULP, CATSUP, PUREE, SAUCE AND PASTE.

27

APPARATUS.

(**a**) *Compound microscope.*—Equipped with apochromatic objectives and compensating oculars, giving magnifications of approximately 90, 180 and 500 diameters. These magnifications can be obtained by the use of 16 and 8 mm. Zeiss apochromatic objectives with X6 and X18 Zeiss compensating oculars, or their equivalents, such as the Spencer 16 and 8 mm. apochromatic objectives with Spencer X10 and X20 compensating oculars, the draw-tube of the microscope being adjusted as directed below.

(**b**) *Thoma-Zeiss blood counting cell.*

(**c**) *Howard mold counting cell.*[1]—Constructed like a blood counting cell but with the inner disk (which need not be ruled) about 19 mm. in diameter.

28

MOLDS.—TENTATIVE.

Clean the special Howard cell so that Newton's rings are produced between the slide and the cover-glass. Remove the cover and place, by means of a knife blade or scalpel, a small drop of the sample upon the central disk; spread the drop evenly over the disk and cover with the cover-glass so as to give an even spread to the material.

It is of the utmost importance that the drop be mixed thoroughly and spread evenly, otherwise the insoluble matter, and consequently the molds, are most abundant at the center of the drop. Squeezing out of the more liquid portions around the margin must be avoided. In a satisfactory mount Newton's rings should be apparent when finally mounted and none of the liquid should be drawn across the moat and under the cover-glass.

Place the slide under the microscope and examine with a magnification of about 90 diameters and with such adjustment that each field of view represents approximately 1.5 sq. mm. of area on the mount. This area is of vital importance and may be obtained by adjusting the draw tube to the proper length as determined by actual measurement of the field, a 16 mm. Zeiss apochromatic objective with a Zeiss X 6 compensating ocular, or a Spencer 16 mm. apochromatic objective with a Spencer X 10 compensating ocular, or their equivalents, being used to obtain the proper magnification.

Observe each field as to the presence or absence of mold filaments and note the result as positive or negative. Examine at least 50 fields, prepared from two or more mounts. No field should be considered positive unless the aggregate length of the filaments present exceeds approximately one-sixth the diameter of the field. Calculate the proportion of positive fields from the results of the examination of all the observed fields and report as percentage of fields containing mold filaments.

29 YEASTS AND SPORES.—TENTATIVE.

Fill a graduated cylinder with water to the 20 cc. mark, and then add the sample till the level of the mixture reaches the 30 cc. mark. Close the graduate, or pour the contents into an Erlenmeyer flask, and shake the mixture vigorously for 15–20 seconds. To facilitate thorough mixing the mixture should not fill more than three-fourths of the container in which the shaking is performed. For tomato sauce or pastes, or products running very high in the number of organisms, or of heavy consistency, 80 cc. of water should be used with 10 cc. or 10 grams of the sample. In the case of exceptionally thick or dry pastes, it may be necessary to make an even greater dilution.

Pour the mixture into a beaker. Thoroughly clean the Thoma-Zeiss counting cell so as to give good Newton's rings. Stir thoroughly the contents of the beaker with a scalpel or knife blade, and then, after allowing to stand 3–5 seconds, remove a small drop and place upon the central disk of the Thoma-Zeiss counting cell and cover immediately with the cover-glass, observing the same precautions in mounting the sample as given under **28**. Allow the slide to stand not less than 10 minutes before beginning to make the count. Make the count with a magnification of about 180 diameters, to obtain which the following combinations, or their equivalents, should be employed: 8 mm. Zeiss apochromatic objective with X 6 Zeiss compensating ocular, or 8 mm. Spencer apochromatic objective with X 10 Spencer compensating ocular with draw-tube not extended.

Count the number of yeasts and spores on one-half of the ruled squares on the disk (this amounts to counting the number in 8 of the blocks, each of which contains 25 of the small ruled squares). The total number thus obtained equals the number of organisms in $\frac{1}{10}$ cmm. if a dilution of 1 part of the sample with 2 parts of water is used. If a dilution of 1 part of the sample with 8 parts of water is used the number must be multiplied by 3. In making the counts, the analyst should avoid counting twice organisms which rest on a boundary line between two adjacent squares.

30 BACTERIA.—TENTATIVE.

Estimate the bacteria from the mounted sample, used in **29**, but allow the sample to stand not less than 15 minutes after mounting before making the count. Employ a magnification of about 500 diameters, which may be obtained by the use of an 8 mm. Zeiss apochromatic objective with X 18 Zeiss compensating ocular with draw-tube not extended, or an 8 mm. Spencer apochromatic objective with X 20 Spencer compensating ocular with a tube length of 190, or their equivalents.

Count and record the number of bacteria in a small area consisting of five of the small sized squares. Move the slide to another portion of the field and count the number on another similar area. Count five such areas, preferably one from near each corner of the ruled portion of the slide and one from near the center. Determine the average number of bacteria per area and multiply by 2,400,000, which gives the number of bacteria per cc. If a dilution of 1 part of the sample with 8 parts of water instead of 1 part of the sample with 2 parts of water is used in making up the sample, then the total count obtained as above must be multiplied by 7,200,000. Omit the micrococcus type of bacteria in making the count.

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[1] U. S. Bur. Chem. Bulls. 125 and 151; U. S. Dept. Agr. Bull. 196; U. S. Bur. Chem. Circ. 54; Research Laboratory, National Canners Association, Bull. 2.

[2] U. S. Bur. Chem. Circ. 68, p. 4.

XIV. CEREAL FOODS.

WHEAT FLOUR.

1

MOISTURE.—OFFICIAL.

Determine moisture as directed in **VII, 2.**

2

ASH.—OFFICIAL.

Determine ash as directed in **VII, 4,** using 3–5 grams of flour.

3

CRUDE FAT OR ETHER EXTRACT.—OFFICIAL.

Determine the ether extract as directed in **VII, 10.** With fine flour the addition of an equal weight of clean, dry sand is often necessary.

4

CRUDE FIBER.—OFFICIAL.

Determine crude fiber as directed in **VII, 66.**

5

ACIDITY OF WATER EXTRACT.—TENTATIVE.

Weigh 18 grams of the flour into a 500 cc. Erlenmeyer flask and add 200 cc. of carbon dioxid-free water. Place the flask, loosely stoppered, for an hour in a water bath kept at 40°C., shaking occasionally. Filter upon a dry, folded filter, returning the first 10–15 cc. of the filtrate to the filter. Titrate 100 cc. of the clear filtrate with N/20 sodium hydroxid, using phenolphthalein as an indicator. Each cc. of N/20 sodium hydroxid is equivalent to 0.05 per cent acidity as lactic acid.

6

SUGARS.—TENTATIVE.

Determine reducing sugars and sucrose as directed in **VII, 57** and **58.**

7

PROTEIN.—OFFICIAL.

Determine nitrogen as directed in **I, 18, 21** or **23.** Multiply the percentage of nitrogen by 5.7 to obtain the percentage of protein.

ALCOHOL-SOLUBLE PROTEINS.

8

Method I. (By nitrogen determination).—Tentative.

Transfer 4 grams of the flour to a 150–200 cc. bottle or Erlenmeyer flask and add 100 cc. of 70 per cent alcohol by volume, taking care that none of the material sticks to the bottom of the container. Shake thoroughly 10–12 times at intervals of 30 minutes at room temperature, or shake continuously in a shaking machine for an hour, and then set aside overnight. Shake thoroughly once more and filter through a dry, folded filter, returning the first runnings to the filter until a clear filtrate is obtained. Pipette 50 cc. of the filtrate, equivalent to 2 grams of the sample, into a Kjeldahl flask, dilute with 100 cc. of water to prevent frothing during digestion and determine nitrogen as directed in **I, 18, 21** or **23.**

Method II. (By Polarization).—Tentative.

9 REAGENT.

Millon's reagent.—Dissolve metallic mercury in an equal weight of concentrated nitric acid and dilute the solution with an equal volume of water. The freshly prepared solution must be used.

10 DETERMINATION.

Weigh 15.97 grams of the flour into a 30 cc. flask and add 100 cc. of alcohol (sp. gr. 0.90). Shake at 30 minute intervals for 3 hours and then let stand overnight. Filter through a dry, folded filter and polarize in a 200 mm. tube. Precipitate the proteins in 50 cc. of the filtrate by the addition of 5 cc. of Millon's reagent. Shake, filter and polarize the filtrate in a 200 mm. tube. Multiply the reading in degrees Ventzke by 1.1 to correct for the dilution and deduct the product from the first reading. This difference, multiplied by 0.2, gives the per cent of gliadin[1] nitrogen.

11 PROTEINS SOLUBLE IN 5 PER CENT POTASSIUM SULPHATE SOLUTION.—TENTATIVE.

Weigh 6 grams of the flour into a 200 cc. flask and introduce exactly 100 cc. of 5 per cent potassium sulphate solution. Shake at 30 minute intervals for three hours or, better, agitate at moderate speed in a shaker for one hour, let settle 30 minutes, filter and determine the nitrogen in 50 cc. of the filtrate as directed in **I, 18, 21** or **23**.

12 GLOBULIN AND ALBUMIN (EDESTIN AND LEUCOSIN) AND AMINO NITROGEN.—TENTATIVE.

Weigh 10 grams of the flour into a 500 cc. Erlenmeyer flask, add 250 cc. of 1 per cent sodium chlorid solution, stopper the flask and shake thoroughly. Let stand, with occasional shaking, for three hours, filter through dry paper and evaporate 100 cc. of the filtrate to a small volume in a Kjeldahl digestion flask with 5 cc. of concentrated sulphuric acid. Add the remainder of the sulphuric acid and determine the nitrogen as directed in **I, 18, 21** or **23**. To a second 100 cc. of the filtrate add 5 cc. of 20 per cent phosphotungstic acid solution, shake thoroughly, allow to settle and filter by decantation. Wash slightly with water, concentrate the filtrate with 5 cc. of sulphuric acid in a Kjeldahl flask and determine the amino nitrogen as directed in **I, 18, 21** or **23**. Deduct the amino nitrogen from the nitrogen found in the first fraction to obtain the nitrogen as globulin and albumin[2].

13 GLUTENIN.—TENTATIVE.

Deduct the sum of the potassium sulphate-soluble nitrogen, **11**, and the alcohol-soluble nitrogen, **8**, from the total nitrogen, **7**, and multiply the difference by 5.7.

14 COLD-WATER-SOLUBLE EXTRACT.—TENTATIVE.

Weigh 20 grams of the flour into a 500 cc. Erlenmeyer flask and add gradually 200 cc. of water at a temperature not higher than 10°C., shake vigorously when about 50 cc. of water have been added and continue shaking during the addition of the remainder. Allow to stand at 10°C. for 40 minutes, shaking occasionally. Filter through a large, dry, coarse filter paper, returning the first runnings to the filter until a clear filtrate is obtained. Pipette 20 cc. of the clear filtrate into a tared dish, evaporate to dryness on a steam bath and dry in an oven at 100°C. for periods of 30 minutes to constant weight.

GLUTEN.

15 *Qualitative Test.*[1]—*Tentative.*

Place a very small quantity (about 1.5 mg.) of the flour on a microscope slide, add a drop of water, containing 0.2 gram of water-soluble eosin in 1 liter, and mix by means of a cover-glass, holding the latter at first in such a manner that it is raised slightly above the slide, and taking care that none of the flour escapes from beneath it. Finally allow the cover-glass to rest on the slide and rub it back and forth until the gluten has collected into rolls. The operation should be carried out on a white paper so that the formation of gluten rolls can be noted. Wheat flour, or other flours containing gluten, show by this treatment a copious amount of gluten, which absorbs the eosin with avidity, assuming a carmine color. Rye and corn flour yield only a trace of gluten; buckwheat flour, no appreciable amount. The preparations are best examined with the naked eye, thus gaining an idea of the amount of gluten present. If the flour is coarse, or contains a considerable amount of bran elements, as is true of buckwheat flour and low-grade wheat flour, the test should be made after bolting, as the bran particles and coarse lumps interfere with the formation of gluten rolls.

16 *Quantitative Method.—Tentative.*

Weigh 25 grams of the flour into a cup or porcelain mortar, add sufficient tap water (about 15 cc.) to form a firm dough ball and work into a dough with a spatula or pestle, taking care that none of the material adheres to the utensil employed. Allow the dough to stand in water at room temperature for an hour, then knead gently in a stream of tap water until the starch and all soluble matters are removed. This operation requires approximately 12 minutes and should be performed over bolting cloth or a horsehair sieve. To determine if the gluten is starch-free let 1 or 2 drops of the wash water, obtained by squeezing the gluten, fall into a beaker containing perfectly clear water. If starch is present a cloudiness appears. Allow the gluten thus obtained to stand in water for an hour, then press as dry as possible between the hands, roll into a ball, place in a tared, flat-bottomed dish and weigh as moist gluten. Transfer to an oven, dry to constant weight at 100°C. (about 24 hours), cool and weigh as dry gluten.

CHLORIN.

17 *Qualitative Test. (Chlorin-Bleached Flours).—Tentative.*

Extract 30 grams of the flour with gasoline and allow the solvent to evaporate. A small amount of oil remains. Heat a piece of copper wire in a colorless gas flame until it is black and no longer colors the flame green. Dip the hot end of the wire into the oil and again bring into the flame. If chlorin or bromin has been used as a bleaching agent, a green or blue coloration is produced.

18 *Quantitative Method. (Added Chlorin in Chlorin-Bleached Flour).—Tentative.*

Weigh 20 grams of the flour into a flat-bottomed aluminium dish, 8–10 cm. in diameter, and dry 5 hours in a boiling water or steam oven, transfer, with as little exposure to the air as possible, to a continuous fat extractor, and extract for 16 hours with anhydrous alcohol-free ether, which is also free from chlorin. Transfer the ether extract to a nickel dish and add 25 cc. of a solution containing 25 grams of sodium or potassium hydroxid and 15 grams of sodium nitrate per liter. Place the dish on a steam bath, evaporate to dryness and ignite in a muffle at a dull red heat until the contents are thoroughly charred. Extract the charred mass with 25 cc. of 1 per cent nitric acid and filter. Return the residue to the dish, char and again extract with 25 cc. of 1 per cent nitric acid, filter, wash with hot water, return to the dish and ignite to a white ash. Dissolve the ash in 5 per cent nitric acid and add the solution to the filtrates previously obtained. Determine the chlorin in the combined filtrates either gravimetrically, as directed in **I, 16** (**a**), or volumetrically, as directed in **II, 17**, using N/50 solutions for greater accuracy.

Special precautions should be taken that the air of the laboratory during the entire operation is not contaminated by chlorin or hydrochloric acid fumes and that all reagents employed are as free as possible from chlorin. In all cases a blank determination should be conducted at the same time and a correction introduced if necessary.

NITRITE NITROGEN.—TENTATIVE.

19 REAGENTS.

(**a**) *Sulphanilic acid solution.*—Dissolve 0.5 gram of sulphanilic acid in 150 cc. of 20 per cent acetic acid.

(**b**) *Alpha-naphthylamin hydrochlorid solution.*—Dissolve, by heating, 0.2 gram of the salt in 150 cc. of 20 per cent acetic acid.

(**c**) *Standard nitrite solution.*—Dissolve 0.1097 gram of dry C. P. silver nitrite in about 20 cc. of hot water, add 0.10 gram of C. P. sodium chlorid, shake until the silver chlorid flocculates and make up to 1 liter. Draw off 10 cc. of the clear solution and dilute to 1 liter. Each cc. of the last solution is equivalent to 0.0001 mg. of nitrogen as nitrite. [Cf. **III, 12** (**d**).]

The silver nitrite may be prepared as follows: To a cold solution of about 2 grams of sodium or potassium nitrite in 50 cc. of water, add a solution of silver nitrate so long as a precipitate appears. Decant the liquid and thoroughly wash the precipitate with cold water. Dissolve in boiling water. On cooling, the silver nitrite crystallizes out. Dry the crystals in the dark at ordinary temperature (preferably in a vacuum).

20 DETERMINATION.

(1) Select a series of 100 cc. volumetric flasks of uniform dimensions and color. Place 2 grams of high grade, nitrite-free flour in each; add approximately 70 cc. of nitrite-free water and shake until the flour is thoroughly moistened. Add to these flasks varying amounts of the standard sodium nitrite solution, so that a series of comparison standards will be obtained having a range covering the probable nitrite content of the unknown sample. Reserve one flask for a blank test. In order to avoid making a large series of standards it is well to make a preliminary test to ascertain the approximate nitrite content of the unknown. Where the quantity of nitrite present is small, the nitrite solution in the flasks may be increased by 0.4 cc. each. Where bleaching is excessive, 1 gram of flour may be used throughout, or the standards may be given a wider variation in nitrite content.

To each of two similar flasks add 2 grams of the flour and 90 cc. of water; shake thoroughly and digest all the flasks, including the blank, in a water bath at 40°C. for at least 15 minutes; add 2 cc. each of the sulphanilic acid and alpha-naphthylamin hydrochlorid solutions to each flask, shaking the mixture after the addition of each reagent. Continue the digestion at 40°C. for an additional 20 minutes. The color must be developed in all the flasks under conditions as nearly uniform as possible. Make up to the marks with nitrite-free water and compare the unknown with the series of standards. This may be done in a large, white enameled pan, the effect of the turbidity due to the flour being minimized by the white background. The solutions should be allowed to subside and should not be shaken during comparison; or,

(2) Weigh 20 grams of the flour into a 500 cc. Erlenmeyer flask, add 200 cc. of nitrite-free water, previously warmed to 40°C., and close the flask with a rubber stopper. Shake vigorously for 5 minutes and digest for an hour in a water bath, keeping the temperature of the liquid in the flask at 40°C. and shaking at 10 minute intervals. Finally filter on a dry, nitrite-free, folded filter. Return the first runnings to the filter until a clear filtrate is obtained. Pipette 50 cc. of the filtrate and 50 cc. of the standard nitrite solution into small flasks; add to each, 50 cc. of water, 2 cc. each of the sulphanilic acid and alpha-naphthylamin hydrochlorid solutions, shake and allow to stand an hour to bring out the color. Compare the two solutions in a colorimeter. Divide the height of the column of the standard solution by that of the solution of the sample, to obtain the parts of nitrogen as nitrous acid (free and combined) per million of flour.

21 GASOLINE COLOR VALUE.—TENTATIVE.

Place 20 grams of the flour in a wide-mouthed, glass-stoppered 120 cc. bottle and add 100 cc. of colorless gasoline. Stopper tightly and shake vigorously for 5 minutes. After standing 16 hours, shake again for a few seconds until the flour has been loosened from the bottom of the bottle and thoroughly mixed with the gasoline, then filter immediately on a dry 11 cm. paper into an Erlenmeyer flask, keeping the funnel covered with a watch glass to prevent evaporation. In order to secure a clear filtrate, a certain quantity of the flour should be allowed to pass over onto the paper and the first portion of the filtrate passed through a second time. It will be found convenient to fit the filter paper to the funnel by means of water. Dry thoroughly either by standing overnight in a well-ventilated place or by heating.

Determine the color value of the clear gasoline solution in a Schreiner or similar colorimeter, using for comparison a 0.005 per cent potassium chromate solution. This solution corresponds to a gasoline number of 1.0 and is conveniently prepared by diluting 10 cc. of a 0.5 per cent solution to 1 liter. The colorimeter tube, containing the gasoline solution, should first be adjusted so as to read 50 mm., then the tube containing the standard chromate solution raised or lowered until the shades of yellow in both tubes match. The reading of the chromate solution, divided by the reading of the gasoline solution, gives the gasoline color value. The color value may be determined also in Nessler tubes, using for comparison potassium chromate solutions of various dilutions prepared from a 0.5 per cent solution and filling the tubes in all cases to the height of 50 mm.

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[1] U. S. Bur. Chem. Bull. 152, p. 104.
[2] U. S. Bur. Chem. Bull. 122, p. 54.
[3] Annal. Physik Chemic, 1852, **85**: 161; U. S. Bur. Chem. Bull. 122, p. 217.

XV. WINES.

(Unless otherwise noted express results as grams per 100 cc.)

1 PHYSICAL EXAMINATION.—TENTATIVE.

Note the following: whether the container is "bottle full"; the appearance of the wine, whether it is bright or turbid and whether there is any sediment; condition when opened, whether still, gaseous or carbonated; color and depth of color; odor, whether vinous, acetous, pleasant or foreign; and taste, whether vinous, acetous, sweet, dry or foreign.

2 PREPARATION OF SAMPLE.—OFFICIAL.

If gas is contained in the wine, remove it by pouring the sample back and forth in beakers.

Filter the wine, regardless of appearance, before analysis and determine immediately the specific gravity and such ingredients as alcohol, acids and sugars which are liable to change through exposure.

3 SPECIFIC GRAVITY.—OFFICIAL.

Determine the specific gravity at $\frac{20^\circ \text{ C.}}{4^\circ}$ by means of a pycnometer. Standardize the instrument as follows: Carefully clean the pycnometer by filling it with a saturated solution of chromic acid in concentrated sulphuric acid and allowing it to stand for several hours. Empty the pycnometer and rinse thoroughly with water. Then fill it with recently boiled water previously cooled to 16°–18°C., place in a bath of water cooled to the same temperature and allow the bath to warm slowly to 20°C., adjust the level of the water to the proper point on the pycnometer, put the perforated cap or stopper in place, remove from the bath, wipe dry with a cloth and, after allowing to stand for 15–20 minutes, weigh. Empty the pycnometer, rinse several times with alcohol and then with ether, allow it to become perfectly dry and weigh. Ascertain the weight of contained water at 20°C. by subtracting the weight of the empty pycnometer from its weight when full, and calculate the weight of contained water at 4°C. by multiplying the result by 1.0018 (determined from the respective densities of water at the two temperatures $\frac{1.00000}{0.99823}$)

To determine the specific gravity of the wine at $\frac{20^\circ \text{ C.}}{4^\circ}$ cool it to 16°–18°C., fill the pycnometer with the wine, immerse in a water bath cooled to 16°–18°C., allow the bath to warm slowly to 20°C., adjust the level of the wine to the proper point on the pycnometer, put the perforated cap or stopper in place, wipe dry and weigh in the same manner as in the standardization with water. Subtract the weight of the empty pycnometer from its weight when filled with wine, and divide the difference by the weight of contained water at 4°C. determined above, the quotient being the specific gravity of the wine at $\frac{20^\circ \text{ C.}}{4^\circ}$

4 ALCOHOL.—OFFICIAL.

(a) *By volume.*—Measure 100 cc. of the liquid at 20°C. into a 300–500 cc. distillation flask and add 50 cc. of water, attach the flask to a vertical condenser by means of a bent tube and distil almost 100 cc., making up to 100 cc. volume when cooled to 20°C. Foaming, which sometimes occurs, especially with young wines, may be prevented by the addition of a small amount of tannin. To determine the alcohol in wines which have undergone acetous fermentation and contain an abnormal amount of acetic acid, exactly neutralize the portion taken with sodium hydroxid solution before distilling. This is unnecessary, however, in wines of normal taste and odor. Determine the specific gravity of the distillate at $\frac{20^\circ \text{ C.}}{4^\circ}$ and obtain the corresponding percentage of alcohol by volume from **XXX**, Table 7.

(**b**) *Grams per 100 cc.*—From the specific gravity of the distillate, obtained in (**a**), ascertain from **XXX**, Table 7, the corresponding alcohol content in grams per 100 cc.

(**c**) *By weight.*—Divide the number of grams in the 100 cc. of distillate, as obtained in (**b**), by the weight of the sample as calculated from its specific gravity.

(**d**) *By immersion refractometer.*—The percentages of alcohol, as determined in (**a**) and (**c**), may be verified by determining the immersion refractometer reading of the distillate and obtaining, from **XXX**, Table 8, the corresponding percentages of alcohol.

GLYCEROL IN DRY WINES.

5

Method I. (By Direct Weighing).—Official.

Evaporate 100 cc. of the wine in a porcelain dish on the water bath to a volume of about 10 cc. and treat the residue with about 5 grams of fine sand and 4–5 cc. of milk of lime (containing about 15 per cent of calcium oxid) for each gram of extract present and evaporate almost to dryness. Treat the moist residue with 50 cc. of 90 per cent alcohol by volume, remove the substance adhering to the sides of the dish with a spatula and rub the whole mass to a paste. Heat the mixture on a water bath, with constant stirring, to incipient boiling and decant the liquid through a filter into a small flask. Wash the residue repeatedly by decantation with 10 cc. portions of hot 90 per cent alcohol until the filtrate amounts to about 150 cc. Evaporate the filtrate to a sirupy consistency in a porcelain dish on a hot, but not boiling, water bath; transfer the residue to a small, glass-stoppered, graduated cylinder with 20 cc. of absolute alcohol and add three portions of 10 cc. each of anhydrous ether, shaking thoroughly after each addition. Let stand until clear, then pour off through a filter, and wash the cylinder and filter with a mixture of 2 parts of absolute alcohol to 3 parts of anhydrous ether, also pouring the wash liquor through the filter. Evaporate the filtrate to a sirupy consistency, dry for an hour at the temperature of boiling water, weigh, ignite and weigh again. The loss on ignition gives the weight of glycerol.

6

Method II. (By Oxidation with Dichromate).—Official.

Evaporate 100 cc. of the wine in a porcelain dish on a water bath, the temperature of which is maintained at 85°–90°C., to a volume of 10 cc. and treat the residue with about 5 grams of fine sand and 5 cc. of milk of lime (containing 15 grams of calcium oxid per 100 cc.). Proceed from this point as directed under **XVIII**, **6**, beginning with the clause "Evaporate almost to dryness with frequent stirring", except that the solution of glycerol after treatment with silver carbonate and lead acetate is made up to 100 cc. instead of 50 cc. Observe the precautions given concerning the temperature at which all evaporations are to be made.

7

GLYCEROL IN SWEET WINES.—OFFICIAL.

With wines whose extract exceeds 5 grams per 100 cc., heat 100 cc. to boiling in a flask and treat with successive small portions of milk of lime until the wine becomes first darker and then lighter in color. Cool, add 200 cc. of 95 per cent alcohol by volume, allow the precipitate to subside, filter and wash with 95 per cent alcohol. Treat the combined filtrate and washings as directed in **5** or **6**.

8 GLYCEROL-ALCOHOL RATIO.—OFFICIAL.

Express this ratio as X : 100, in which X is obtained by multiplying the percentage weight of glycerol by 100 and dividing the result by the percentage of alcohol by weight.

EXTRACT.

9 *From the Specific Gravity of the Dealcoholized Wine.—Official.*

Calculate the specific gravity of the dealcoholized wine by the following formula:

$$S = G + 1 - A \text{ in which}$$

S = specific gravity of the dealcoholized wine;
G = specific gravity of the wine, **3**; and
A = specific gravity of the distillate obtained in the determination of alcohol, **4** **(a)**.

From **XXX**, Table 3, ascertain the per cent by weight of extract in the dealcoholized wine corresponding to the value of S. Multiply the figure thus obtained by the value of S to obtain the grams of extract per 100 cc. of wine.

10 *By Evaporation.—Official.*

(a) *In dry wines, having an extract content of less than 3 grams per 100 cc.*—Evaporate 50 cc. of the sample on a water bath to a sirupy consistency in a 75 cc. flat-bottomed platinum dish, approximately 85 mm. in diameter. Heat the residue for 2–5 hours in a drying oven at the temperature of boiling water, cool in a desiccator and weigh as soon as the dish and contents reach room temperature.

(b) *In sweet wines.*—When the extract content is between 3 and 6 grams per 100 cc.; treat 25 cc. of the sample as directed under **(a)**.

When the extract exceeds 6 grams per 100 cc., however, the result, obtained as directed under **9**, is accepted and no gravimetric determination is attempted. This is because of the serious error connected with drying levulose at high temperature.

11 NON-SUGAR SOLIDS.—OFFICIAL.

Determine the non-sugar solids (sugar-free extract) by subtracting the amount of reducing sugars before inversion, **12**, from the extract, **9** or **10**. If sucrose is present in the wine, determine the non-sugar solids by subtracting the sum of reducing sugars before inversion and the sucrose from the extract.

12 REDUCING SUGARS.—OFFICIAL.

(a) *Dry Wines.*—Place 200 cc. of the wine in a porcelain dish, exactly neutralize with N/1 sodium hydroxid, calculating the amount required from the determination of acidity, **23**, and evaporate to about one-fourth the original volume. Transfer to a 200 cc. flask, add sufficient neutral lead acetate solution to clarify, dilute to the mark with water, shake and filter through a folded filter. Remove the lead with dry potassium oxalate and determine reducing sugars as directed under **VII, 25**.

(b) *Sweet wines.*—In the case of sweet wines approximate the sugar content by subtracting 2 from the result in the determination of the extract and employ such a quantity of the sample that the aliquot taken for the copper reduction shall not exceed 240 mg. of invert sugar. Proceed as directed in **(a)** except that this smaller quantity of the sample is taken for the determination.

SUCROSE.

13 *By Reducing Sugars Before and After Inversion.—Official.*

Proceed as directed under **VII, 18**, using the method given under **VII, 25**, for the determination of reducing sugars.

14 *By Polarization.—Official.*

Polarize part of the filtrate, obtained in **12**, before and after inversion in a 200 mm. tube as directed under **VII, 14** or **16**. In calculating the percentage of sucrose the relation of the amount of sample contained in 100 cc. to the normal weight for the instrument must be taken into consideration.

15 COMMERCIAL GLUCOSE.—OFFICIAL.

Polarize a portion of the filtrate, obtained in **12**, after inversion in a 200 mm. jacketed tube at 87°C. as directed under **VIII, 22**. In calculating the percentage of glucose the relation of the amount of sample contained in 100 cc. to the normal weight for the instrument must be taken into consideration.

16 ASH.—OFFICIAL.

Proceed as directed under **VII, 4**, employing the residue from 50 cc. of the wine.

17 ASH-EXTRACT RATIO.—OFFICIAL.

Express results as 1 : X, in which X is the quotient obtained by dividing the grams of extract per 100 cc. by the grams of ash per 100 cc.

18 ALKALINITY OF THE WATER-SOLUBLE ASH.—OFFICIAL.

Extract the ash, obtained as directed under **16**, with successive small portions of hot water until the filtrate amounts to about 60 cc. and proceed as directed under **VIII, 15**. Express the alkalinity in terms of the number of cc. of N/10 acid required to neutralize the water-soluble ash from 100 cc. of the wine.

19 ALKALINITY OF THE WATER-INSOLUBLE ASH.—OFFICIAL.

Ignite the filter and residue from **18** in the platinum dish in which the wine was ashed, and proceed as directed under **VIII, 16**. Express the alkalinity in terms of the number of cc. of N/10 acid required to neutralize the water-insoluble ash from 100 cc. of the wine.

20 PHOSPHORIC ACID.—OFFICIAL.

Dissolve the ash, obtained as directed under **16**, in 50 cc. of boiling nitric acid (1 to 9), filter, wash the filter and determine phosphoric acid in the combined filtrate and washings, as directed in **I, 6** or **9**. If the ash ignites without difficulty, no free phosphoric acid need be suspected. Should there be any free acid, the ash remains black even after repeated leaching. In such cases calcium acetate or a mixture containing 3 parts of sodium carbonate and 1 of sodium nitrate should be added to avoid loss of phosphoric acid before attempting to ash.

21 SULPHURIC ACID.—OFFICIAL.

Precipitate directly the sulphuric acid in 50 cc. of the wine by means of barium chlorid solution, after acidifying with a small excess of hydrochloric acid, and determine the resulting barium sulphate as directed under **II, 12**. Allow the precipitate to stand for at least 6 hours before filtering. Report as sulphur trioxid (SO_3), using the factor 0.3430.

22 CHLORIDS.—OFFICIAL.

To 100 cc. of dry wine or 50 cc. of sweet wine add sufficient sodium carbonate to make distinctly alkaline. Evaporate to dryness, ignite at a heat not above low redness, cool, extract the residue with hot water, acidify the water extract with nitric acid and determine chlorids as directed under **II, 15** or **17**.

23 TOTAL ACIDS.—OFFICIAL.

Measure 20 cc. of the wine into a 250 cc. beaker, heat rapidly to incipient boiling and immediately titrate with N/10 sodium hydroxid. Determine the end point with neutral 0.05 per cent azolitmin solution as an outside indicator. Place the indicator in the cavities of a spot plate and spot the wine into the azolitmin solution. The end point is reached when the color of the indicator remains unchanged by the addition of a few drops of N/10 alkali to the wine.

In the case of wines which are artificially colored and which can not be satisfactorily titrated in the above manner, it will be found helpful to use phenolphthalein powder (1 part of phenolphthalein mixed with 100 parts of dry, powdered potassium sulphate) as an indicator. Place this indicator in the cavities of a spot plate and spot the wine into the powder. The end of the titration is indicated when the powder acquires a pink tint.

Express the result in terms of tartaric acid. One cc. of N/10 sodium hydroxid is equivalent to 0.0075 gram of tartaric acid.

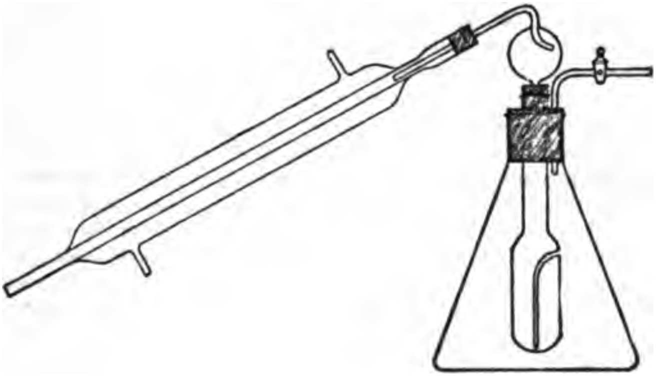

FIG. 6. APPARATUS FOR THE DETERMINATION OF VOLATILE ACIDS.

VOLATILE ACIDS.

24 *Method I.—Official.*

Heat rapidly to incipient boiling 50 cc. of the wine in a 500 cc. distillation flask and pass steam through until 15 cc. of the distillate require only 2 drops of N/10 sodium hydroxid for neutralization. The water used to generate the steam should be boiled several minutes before connecting the steam generator with the distillation flask in order to expel carbon dioxid. Titrate rapidly with N/10 sodium hydroxid, using phenolphthalein as an indicator. The color should remain about 10 seconds. Express the result as acetic acid. One cc. of N/10 sodium hydroxid is equivalent to 0.0060 gram of acetic acid.

25 *Method II*[1]*.—Official.*

Introduce 10 cc. of the wine, previously freed from carbon dioxid, into the inner tube of a modified Sellier distillation apparatus (Fig. 6), add a small piece of paraffin to prevent foaming, and adjust the tube and its contents in place within the larger flask containing 100 cc. of recently boiled water. Connect with a condenser as illustrated in Fig. 6 and distil by heating the outer flask. When 50 cc. of the distillate have been collected, empty the receiver into a beaker and titrate with N/10 sodium hydroxid, using phenolphthalein as an indicator. Continue the distillation and titrate each succeeding 10 cc. of distillate until not more than 1 drop of standard alkali is required to reach the neutral point. Usually 80 cc. of distillate will contain all the volatile acids.

26

FIXED ACIDS.—OFFICIAL.

Multiply the amount of volatile acids by 1.25 and subtract this from the total acids, to obtain the amount of fixed acids, expressed as tartaric acid.

27

TOTAL TARTARIC ACID[2].—OFFICIAL.

Neutralize 100 cc. of the wine with N/1 sodium hydroxid, calculating from the acidity, **23**, the number of cc. of N/1 alkali necessary for the neutralization. If the volume of the solution is increased more than 10 per cent by the addition of the alkali, evaporate to approximately 100 cc. Add to the neutralized solution 0.075 gram of tartaric acid for each cc. of N/1 alkali added and, after the tartaric acid has dissolved, add 2 cc. of glacial acetic acid and 15 grams of potassium chlorid. After the potassium chlorid has dissolved, add 15 cc. of 95 per cent alcohol by volume, stir vigorously until the potassium bitartrate begins to precipitate and then let stand in an ice box for at least 15 hours. Decant the liquid from the separated potassium bitartrate on a Gooch crucible prepared with a very thin film of asbestos, or on filter paper in a Büchner funnel. Wash the precipitate and filter 3 times with a few cc. of a mixture of 15 grams of potassium chlorid, 20 cc. of 95 per cent alcohol by volume and 100 cc. of water, using not more than 20 cc. of the wash solution in all. Transfer the asbestos or paper and precipitate to the beaker in which the precipitation was made, wash out the Gooch crucible or Büchner funnel with hot water, using about 50 cc. in all, heat to boiling and titrate the hot solution with N/10 sodium hydroxid, using phenolphthalein as an indicator. Increase the number of cc. of N/10 alkali required by 1.5 cc. to allow for the solubility of the precipitate. One cc. of N/10 alkali is equivalent, under these conditions, to 0.015 gram of tartaric acid. Subtract the amount of tartaric acid added from this result to obtain the grams of total tartaric acid per 100 cc. of the wine.

28

FREE TARTARIC ACID AND CREAM OF TARTAR[3].—OFFICIAL.

Calculate the free tartaric acid and cream of tartar in the following manner:

Let A = total tartaric acid in 100 cc. of wine, divided by 0.015;
B = total alkalinity of the ash (sum of C and D);
C = alkalinity of water-soluble ash;
D = alkalinity of water-insoluble ash.

Then

(1) If A is greater than B,
Cream of tartar = 0.0188 × C; and
Free tartaric acid = 0.015 × (A–B).

(2) If A equals B or is smaller than B but greater than C,
Cream of tartar = 0.0188 × C; and
Free tartaric acid = 0.

(3) If A is smaller than C,
Cream of tartar = 0.0188 × A; and
Free tartaric acid = 0.

TANNIN AND COLORING MATTER.—OFFICIAL.

29 REAGENTS.

(a) *N/10 oxalic acid.*—One cc. is equivalent to 0.00416 gram of tannin.

(b) *Standard potassium permanganate solution.*—Dissolve 1.333 grams of potassium permanganate in 1 liter of water and standardize the solution against (a).

(c) *Indigo solution.*—Dissolve 6 grams of sodium sulphindigotate in 500 cc. of water by heating; cool, add 50 cc. of concentrated sulphuric acid, make up to 1 liter and filter.

(d) *Purified boneblack*[4].—Boil 100 grams of finely powdered boneblack with successive portions of hydrochloric acid (1 to 3), filter and wash with boiling water until free from chlorids. Keep covered with water.

30 DETERMINATION[5].

Dealcoholize 100 cc. of the wine by evaporation and dilute with water to the original volume. Transfer 10 cc. to a 2 liter porcelain dish; add about a liter of water and exactly 20 cc. of the indigo solution. Add the standard potassium permanganate solution, 1 cc. at a time, until the blue color changes to green, then add a few drops at a time until the color becomes golden yellow. Designate the number of cc. of permanganate solution used as "a".

Treat 10 cc. of the dealcoholized wine, prepared as above, with boneblack for 15 minutes; filter and wash the boneblack thoroughly with water. Add a liter of water and 20 cc. of the indigo solution and titrate with permanganate as above. Designate the number of cc. of permanganate used as "b".

Then $a-b=c$, the number of cc. of the permanagante solution required for the oxidation of the tannin and coloring matter in 10 cc. of the wine.

31 CRUDE PROTEIN.—OFFICIAL.

Determine nitrogen in 50 cc. of the wine, as directed under **I, 18, 21** or **23**, and multiply the result by 6.25.

32 PENTOSANS.—OFFICIAL.

Proceed as directed in **VII, 63**, except that 100 cc. of the wine and 43 cc. of hydrochloric acid (sp. gr. 1.19) are used in beginning the distillation. Owing to the interference of sugars this determination can be made in dry wines only.

33 GUM AND DEXTRIN.—TENTATIVE.

Evaporate 100 cc. of the wine to about 10 cc. and add 10 cc. of 95 per cent alcohol by volume. If gum or dextrin be present (indicated by the formation of a voluminous precipitate), continue the addition of alcohol, slowly and with stirring, until 100 cc. have been added. Let stand overnight, filter, and wash with 80 per cent alcohol by volume. Dissolve the precipitate on the paper with hot water, hydrolyze the filtrate and washings with hydrochloric acid and proceed as directed under **VII, 59**.

34 NITRATES.—TENTATIVE.

(a) *White wine.*—Treat a few drops of the wine in a porcelain dish with 2–3 cc. of concentrated sulphuric acid, which contains about 0.1 gram of diphenylamin[6] per 100 cc. The deep blue color formed in the presence of nitrates appears so quickly that it is not obscured, even in sweet wine, by the blackening produced by the action of sulphuric acid on the sugar.

(b) *Red wine.*—Clarify with basic lead acetate, filter, remove the excess of lead from the filtrate with sodium sulphate, filter again and treat a few drops of this filtrate as directed under (a).

35 COLORING MATTERS.—TENTATIVE.

Proceed as directed under X.

36 PRESERVATIVES.—OFFICIAL.

Proceed as directed under IX.

The detection of added boric acid is somewhat difficult because a small amount of it is normally present in certain wines. Therefore, a quantitative determination should be made. The determination of sulphurous acid must also be quantitative. A small amount of salicylic acid is also normal in wine, and for that reason not more than 50 cc. of the sample should be used in testing for that preservative.

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[3] Ibid., p. 75.
[4] U. S. P., VIII, 1907, p. 89.
[5] Ann. Oenologie, 1871–72, 2: 1.
[6] Arch. Hyg., 1884, 2: 273.

XVI. DISTILLED LIQUORS.

1

SPECIFIC GRAVITY.—OFFICIAL.

Determine the specific gravity at $\frac{20° C.}{4°}$ by means of a pycnometer, as directed under XV, 3, or by means of a small, accurately graduated hydrometer.

2

ALCOHOL BY WEIGHT.—OFFICIAL.

Weigh 20–25 grams of the sample into a distillation flask, dilute with 100 cc. of water, distil nearly 100 cc. and weigh the distillate or make to volume at 20°C. and, in either case, determine the specific gravity as directed under 1. Obtain the corresponding percentage of alcohol by weight from XXX, Table 7, multiply this figure by the weight of the distillate, and divide by the weight of the sample taken to obtain the per cent of alcohol by weight.

The alcohol content of the distillate may be checked by determining the immersion refractometer reading and obtaining, from XXX, Table 8, the percentage of alcohol.

ALCOHOL BY VOLUME.

3

Method I.—Official.

From the specific gravity of the distillate, obtained under 2, ascertain the corresponding percentage of alcohol by volume from XXX, Table 7. Multiply this figure by the volume of distillate and divide by the volume of the sample (calculated from the specific gravity) to obtain the percentage of alcohol by volume in the original sample.

4

Method II.—Official.

Measure 25 cc. of the sample at 20°C. into a distillation flask, dilute with 100 cc. of water, distil nearly 100 cc., make to volume at 20°C. and determine the specific gravity as directed in 1. Obtain, from XXX, Table 7, the corresponding percentage of alcohol by volume in the distillate. Multiply by 4 to obtain the percentage of alcohol by volume in the original substance.

The alcohol content of the distillate may be checked by determining the immersion refractometer reading and obtaining the percentage of alcohol from XXX, Table 8.

5

EXTRACT.—OFFICIAL.

Weigh, or measure at 20°C., 100 cc. of the sample, evaporate nearly to dryness on a water bath, then transfer to a water oven, and dry at the temperature of boiling water for 2.5 hours.

6

ASH.—OFFICIAL.

Proceed as directed under VII, 4, employing the residue from the determination of the extract, 5.

7

ACIDITY.—OFFICIAL.

Titrate 100 cc. of the sample (or 50 cc. diluted to 100 cc. if the sample is dark) with N/10 alkali, using phenolphthalein as an indicator. Express the result as acetic acid; 1 cc. of N/10 alkali is equivalent to 0.0060 gram of acetic acid.

8 ESTERS.—OFFICIAL.

Measure 200 cc. of the sample into a distillation flask, add 25 cc. of water and distil slowly 200 cc., using a mercury valve to prevent loss of alcohol. Exactly neutralize the free acid in 50 cc. of the distillate with N/10 alkali, add a measured excess of 25–50 cc. of N/10 alkali, and either boil for an hour under a reflux condenser, cool and titrate with N/10 acid, or allow the solution to stand overnight in a stoppered flask with the excess of alkali, heat with a tube condenser for 30 minutes at a temperature below the boiling point, cool and titrate. Calculate the number of cc. of N/10 alkali used in the saponification of the esters as ethyl acetate; 1 cc. of N/10 alkali is equivalent to 0.0088 gram of ethyl acetate. Run a blank, using water in place of the distillate, and make any necessary correction.

ALDEHYDES.—OFFICIAL.

9 REAGENTS.

(**a**) *Aldehyde-free alcohol.*—Redistil 95 per cent alcohol over sodium or potassium hydroxid, then add 2–3 grams per liter of meta-phenylendiamin hydrochlorid, digest at ordinary temperature for several days (or under a reflux condenser on a steam bath for several hours) and then distil slowly, rejecting the first 100 cc. and the last 200 cc. of the distillate.

(**b**) *Sulphite-fuchsin solution.*—Dissolve 0.50 gram of pure fuchsin in 500 cc. of water, then add 5 grams of sulphur dioxid dissolved in water, make up to 1 liter and allow to stand until colorless. This solution does not keep indefinitely; therefore, prepare it in small quantities and keep at a low temperature.

(**c**) *Standard acetaldehyde solution.*—Prepare according to the directions of Vasey[1] as follows: Grind aldehyde ammonia in a mortar with anhydrous ether and decant the ether. Repeat this operation several times, then dry the purified salt in a current of air and finally in vacuo over sulphuric acid. Dissolve 1.386 grams of this purified aldehyde ammonia in 50 cc. of 95 per cent alcohol by volume, add 22.7 cc. of N/1 alcoholic sulphuric acid, then make up to 100 cc. and add 0.8 cc. of alcohol for the volume of the ammonium sulphate precipitate. Allow the mixture to stand overnight and filter. This solution contains 1 gram of acetaldehyde in 100 cc. and will retain its strength.

The standard found most convenient for use is 2 cc. of this strong aldehyde solution diluted to 100 cc. with 50 per cent alcohol by volume. One cc. of this solution is equivalent to 0.0002 gram of acetaldehyde. This solution should be made up fresh every day or so, as it loses strength.

10 DETERMINATION.

Determine the aldehyde in the distillate, prepared as directed under **8**. Dilute 5–10 cc. of the distillate to 50 cc. with aldehyde-free alcohol (50 per cent by volume), add 25 cc. of the sulphite-fuchsin solution and allow to stand for 15 minutes at 15°C. The solutions and reagents should be at 15°C. when they are mixed. Prepare standards of known strength and blanks in the same way. The comparison standards found most convenient for use contain 0.0001, 0.0002, 0.0003, 0.0004, 0.0005 and 0.0006 gram of acetaldehyde.

FURFURAL.—OFFICIAL.

11 REAGENT.

Standard furfural solution.—Dissolve 1 gram of redistilled furfural in 100 cc. of 95 per cent alcohol by volume. Standards are made by diluting 1 cc. of this solution to 100 cc. with 50 per cent alcohol by volume. One cc. of this solution contains 0.0001 gram of furfural. The strong furfural solution will retain its strength but the dilute solution will not.

12

DETERMINATION.

Dilute 10–20 cc. of the distillate, as prepared under 8, to 50 cc. with furfural-free alcohol, 50 per cent by volume. Add 2 cc. of colorless anilin and 0.5 cc. of hydrochloric acid (sp. gr. 1.125) and keep for 15 minutes in a water bath at about 15°C. Prepare standards of known strength and blanks in the same way. The comparison standards found most convenient for use contain 0.00005, 0.0001, 0.00015, 0.0002, 0.00025 and 0.0003 gram of furfural.

FUSEL OIL.—OFFICIAL.

13

REAGENTS.

(a) *Purified carbon tetrachlorid.*—Mix crude carbon tetrachlorid with one-tenth its volume of concentrated sulphuric acid, shake thoroughly at frequent intervals and allow to stand overnight. Wash free of acid and impurities with tap water. Remove the water, add an excess of sodium hydroxid solution and distil the carbon tetrachlorid from it.

The refuse carbon tetrachlorid after titration is purified for further work by collecting in a large bottle, adding concentrated sodium hydroxid solution, shaking, then washing with tap water until the washings are neutral to phenolphthalein and distilling.

(b) *Oxidizing solution.*—Dissolve 100 grams of potassium dichromate in 900 cc. of water and add 100 cc. of concentrated sulphuric acid.

14

DETERMINATION.

(1) To 100 cc. of the sample add 20 cc. of N/2 sodium hydroxid and saponify the mixture by boiling for an hour under a reflux condenser; or, (2) Mix 100 cc. of the liquor with 20 cc. of N/2 sodium hydroxid, allow to stand overnight at room temperature and distil directly. Connect the flask with a distillation apparatus, distil 90 cc., add 25 cc. of water and continue the distillation until an additional 25 cc. are collected.

Whenever aldehydes are present in excess of 15 parts per 100,000, add to the distillate 0.5 gram of meta-phenylendiamin hydrochlorid, boil under a reflux condenser for an hour, distil 100 cc., add 25 cc. of water and continue the distillation until an additional 25 cc. are collected.

Approximately saturate the distillate with finely ground sodium chlorid and add saturated sodium chlorid solution until the specific gravity is 1.10.

Extract this salt solution four times with the purified carbon tetrachlorid, using 40, 30, 20 and 10 cc., respectively, and wash the carbon tetrachlorid three times with 50 cc. portions of saturated sodium chlorid solution, and twice with saturated sodium sulphate solution. Then transfer the carbon tetrachlorid to a flask containing 50 cc. of the oxidizing solution and boil for 8 hours under a reflux condenser.

Add 30 cc. of water and distil until only about 20 cc. remain; add 80 cc. of water and again distil until 15–20 cc. are left. Neutralize the distillate to methyl orange, and titrate with N/10 sodium hydroxid, using phenolphthalein as an indicator. If the distillations have been properly conducted, the distillate will not show a marked acid reaction to methyl orange. Should considerably more than 1 cc. of N/10 alkali be consumed at this point, the result will be unreliable and the determination should be repeated. One cc. of N/10 sodium hydroxid is equivalent to 0.0088 gram of amyl alcohol.

Rubber stoppers can be used in the saponification and first distillation, but corks covered with tinfoil must be used in the oxidation and second distillation. Corks and tinfoil must be renewed frequently.

Conduct a blank determination upon 100 cc. of carbon tetrachlorid, beginning the blank at that point of the procedure immediately after the extraction and just before the washings with sodium chlorid and sodium sulphate solutions.

15 SUGARS.—OFFICIAL.

Proceed as directed under **XV, 13, 14** or **15.**

METHYL ALCOHOL.

16 *Trillat Method*[2].—*Official.*

To 50 cc. of the sample add 50 cc. of water and 8 grams of lime and fractionate by the aid of Glinsky bulb tubes. Dilute the first 15 cc. of the distillate to 150 cc., mix with 15 grams of potassium dichromate and 70 cc. of sulphuric acid (1 to 5) and allow to stand for an hour with occasional shaking.

Distil, reject the first 25 cc. and collect 100 cc. Mix 50 cc. of the distillate with 1 cc. of redistilled dimethylanilin, transfer to a stout, tightly stoppered flask and keep on a bath at 70°–80°C. for 3 hours with occasional shaking. Make distinctly alkaline with sodium hydroxid solution, and distil off the excess of dimethylanilin, stopping the distillation when 25 cc. have passed over.

Acidify the residue in the flask with acetic acid, shake and test a few cc. by adding 4 or 5 drops of a 1 per cent suspension of lead dioxid. If methyl alcohol is present, a blue coloration occurs which is increased by boiling.

Ethyl alcohol thus treated yields a blue coloration changing immediately to green, later to yellow, and becoming colorless when boiled.

17 *Riche and Bardy Method*[3].—*Official.*

The following method for the detection of methyl alcohol in commercial spirit of wine depends on the formation of methylanilin violet:

Place 10 cc. of the sample, previously redistilled over potassium carbonate if necessary, in a small flask with 15 grams of iodin and 2 grams of red phosphorus. Keep in ice water for 10–15 minutes until action has ceased. Distil off, on a water bath, the methyl and ethyl iodids formed into about 30 cc. of water. Wash with dilute alkali to eliminate free iodin. Separate the heavy, oily liquid which settles and transfer to a flask containing 5 cc. of anilin. If the action be too violent, place the flask in cold water; if too slow, stimulate by gently warming the flask. After an hour boil the product with water, cool and add about 20 cc. of 15 per cent sodium hydroxid solution; when the bases rise to the top as an oily layer, fill the flask up to the neck with water and draw them off with a pipette. Oxidize 1 cc. of the oily liquid by adding 10 grams of a mixture of 100 parts of clean sand, 2 of common salt, and 3 of cupric nitrate, mix thoroughly, transfer to a glass tube and heat to 90°C. for 8–10 hours. Exhaust the product with warm alcohol, filter and make up to 100 cc. with alcohol. If the sample of spirits is pure, the liquid has a red tint, but, in the presence of 1 per cent of methyl alcohol, it has a distinct violet shade; with 2.5 per cent the shade is very distinct, and still more so with 5 per cent. To detect more minute quantities of methyl alcohol, dilute 5 cc. of the colored liquid to 100 cc. with water, and dilute 5 cc. of this again to 400 cc. Heat the liquid thus obtained in a porcelain dish and immerse in it a fragment of white merino (free from sulphur) for 30 minutes. If the alcohol is pure, the wool will remain white, but, if methyl alcohol is present, the fiber will become violet, the depth of tint giving a fairly approximate indication of the proportion of methyl alcohol present.

18 *Immersion Refractometer Method*[4].—*Official.*

Determine by the immersion refractometer at 20°C. the refraction of the distillate obtained in the determination of alcohol. If, on reference to the table under **19**, the refraction shows the percentage of alcohol agreeing with that obtained from the specific gravity, it may safely be assumed that no methyl alcohol is present. If, however, there is an appreciable amount of methyl alcohol, the low refractometer reading will at once indicate the fact. If the absence from the solution of refractive substances other than water and the alcohols is assured, this difference in refraction is conclusive of the presence of methyl alcohol.

The addition of methyl alcohol to ethyl alcohol decreases the refraction in direct proportion to the amount present; hence the quantitative calculation is readily made by interpolation in the table under 19, using the figures for pure ethyl and methyl alcohol of the same alcoholic strength as the sample.

Example.—The distillate has a specific gravity of 0.97080, corresponding to 18.38 per cent alcohol by weight, and has a refraction of 35.8 at 20°C. by the immersion refractometer; by interpolation in the refractometer table the readings of ethyl and methyl alcohol corresponding to 18.38 per cent alcohol are 47.3 and 25.4, respectively, the difference being 21.9; $47.3-35.8=11.5$; $(11.5\div21.9)\,100=52.5$, showing that 52.5 per cent of the total alcohol present is methyl alcohol.

19

Table 11.

Scale readings on Zeiss immersion refractometer at 20°C., corresponding to each per cent by weight of methyl and ethyl alcohols.

PER CENT ALCOHOL BY WEIGHT	SCALE READINGS		PER CENT ALCOHOL BY WEIGHT	SCALE READINGS		PER CENT ALCOHOL BY WEIGHT	SCALE READINGS		PER CENT ALCOHOL BY WEIGHT	SCALE READINGS	
	Methyl alcohol	Ethyl alcohol		Methyl alcohol	Ethyl alcohol		Methyl alcohol	Ethyl alcohol		Methyl alcohol	Ethyl alcohol
0	14.5	14.5	25	29.7	60.1	50	39.8	90.3	75	29.7	101.0
1	14.8	16.0	26	30.3	61.9	51	39.7	91.1	76	29.0	101.0
2	15.4	17.6	27	30.9	63.7	52	39.6	91.8	77	28.3	100.9
3	16.0	19.1	28	31.6	65.5	53	39.6	92.4	78	27.6	100.9
4	16.6	20.7	29	32.2	67.2	54	39.5	93.0	79	26.8	100.8
5	17.2	22.3	30	32.8	69.0	55	39.4	93.6	80	26.0	100.7
6	17.8	24.1	31	33.5	70.4	56	39.2	94.1	81	25.1	100.6
7	18.4	25.9	32	34.1	71.7	57	39.0	94.7	82	24.3	100.5
8	19.0	27.8	33	34.7	73.1	58	38.6	95.2	83	23.6	100.4
9	19.6	29.6	34	35.2	74.4	59	38.3	95.7	84	22.8	100.3
10	20.2	31.4	35	35.8	75.8	60	37.9	96.2	85	21.8	100.1
11	20.8	33.2	36	36.3	76.9	61	37.5	96.7	86	20.8	99.8
12	21.4	35.0	37	36.8	78.0	62	37.0	97.1	87	19.7	99.5
13	22.0	36.9	38	37.3	79.1	63	36.5	97.5	88	18.6	99.2
14	22.6	38.7	39	37.7	80.2	64	36.0	98.0	89	17.3	98.9
15	23.2	40.5	40	38.1	81.3	65	35.5	98.3	90	16.1	98.6
16	23.9	42.5	41	38.4	82.3	66	35.0	98.7	91	14.9	98.3
17	24.5	44.5	42	38.8	83.3	67	34.5	99.1	92	13.7	97.8
18	25.2	46.5	43	39.2	84.2	68	34.0	99.4	93	12.4	97.2
19	25.8	48.5	44	39.3	85.2	69	33.5	99.7	94	11.0	96.4
20	26.5	50.5	45	39.4	86.2	70	33.0	100.0	95	9.6	95.7
21	27.1	52.4	46	39.5	87.0	71	32.3	100.2	96	8.2	94.9
22	27.8	54.3	47	39.6	87.8	72	31.7	100.4	97	6.7	94.0
23	28.4	56.3	48	39.7	88.7	73	31.1	100.6	98	3.5	93.0
24	29.1	58.2	49	39.8	89.5	74	30.4	100.8	99	3.5	92.0
									100	2.0	91.0

20 COLORING MATTERS.—TENTATIVE.

Proceed as directed under X.

21 WATER-INSOLUBLE COLOR IN WHISKIES.—TENTATIVE.

Evaporate 50 cc. of the sample just to dryness on a steam bath. Take up with cold water, using approximately 15 cc., filter and wash until the filtrate amounts to nearly 25 cc. To this filtrate add 25 cc. of absolute alcohol, or 26.3 cc. of 95 per cent alcohol by volume, and make up to 50 cc. with water. Mix thoroughly and compare in a colorimeter with the original material. Calculate from these readings the per cent of color insoluble in water.

22 COLORS INSOLUBLE IN AMYL ALCOHOL.—TENTATIVE.

Evaporate 50 cc. of whisky just to dryness on a steam bath. Dissolve the residue in water and 95 per cent alcohol by volume and make to a volume of 50 cc., using a total volume of 26.3 cc. of 95 per cent alcohol. Place 25 cc. of this solution in a separatory funnel and add 20 cc. of freshly shaken Marsh reagent (100 cc. of pure amyl alcohol, 3 cc. of sirupy phosphoric acid and 3 cc. of water), shaking lightly so as not to form an emulsion. Allow the layers to separate and repeat this shaking and standing twice again. After the layers have separated completely draw off the lower or aqueous layer, which contains the caramel, into a 25 cc. cylinder and make up to volume with 50 per cent alcohol by volume. Compare this solution in a colorimeter with the untreated 25 cc. Calculate the result of this reading to the per cent of color insoluble in amyl alcohol.

23 CARAMEL[5].—TENTATIVE.

Add 10 cc. of paraldehyde to 5 cc. of the sample in a test tube and shake. Add absolute alcohol, a few drops at a time, shaking after each addition until the mixture becomes clear. Allow to stand. Turbidity after 10 minutes is an indication of caramel.

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XVII. BEERS.

(Unless otherwise noted, express results as grams per 100 cc.)

1

PREPARATION OF SAMPLE.—OFFICIAL.

Remove carbon dioxid by transferring the sample to a large flask and shaking vigorously or by pouring back and forth between beakers, care being taken that the temperature of the beer is not below 20°C.

2

COLOR.—TENTATIVE.

Determine the depth of color of the sample in a ½ inch cell with a Lovibond tintometer, using the beer scale. Express the result in terms of a ½ inch cell.

3

SPECIFIC GRAVITY.—OFFICIAL.

Determine the specific gravity at $\frac{20^\circ \text{C.}}{4^\circ}$ by means of a pycnometer as directed under **XV**, 3.

4

ALCOHOL.—OFFICIAL.

Determine as directed under **XV**, 4.

EXTRACT.

5

Method I.—Official.

Measure 25 cc. of the carbon dioxid-free beer at 20°C. into a tared, flat-bottomed platinum dish, approximately 85 mm. in diameter, evaporate just to dryness on a steam bath and heat to constant weight in a vacuum oven at 70°C.

6

Method II.—Tentative.

The immersion refractometer reading of the beer at 20°C., minus the immersion refractometer reading of the distillate at 20°C. times 0.2571, equals the grams of extract in 100 cc. of beer.

7

Method III.—Official.

Calculate the specific gravity of the dealcoholized beer by the following formula:

$$S = G + 1 - A$$ in which

S = the specific gravity of the dealcoholized beer;
G = the specific gravity of the beer; and
A = the specific gravity of the distillate obtained in the determination of alcohol.

From **XXX**, Table 3, ascertain the per cent by weight of extract in the dealcoholized beer corresponding to the value of S. Multiply the figure thus obtained by S to obtain the grams of extract per 100 cc. of beer.

8

EXTRACT OF ORIGINAL WORT (APPROXIMATE).—OFFICIAL.

Calculate the grams of extract per 100 cc. in the original wort by the following formula:

$$O = 2A + E$$ in which

O = extract of the original wort;
A = alcohol (grams per 100 cc.); and
E = extract of the dealcoholized beer (grams per 100 cc.).

9 DEGREE OF FERMENTATION.—OFFICIAL.

Calculate the degree of fermentation by the following formula:

$$D = \frac{100 \times 2A}{O} \text{ in which}$$

D = degree of fermentation;
A = alcohol (grams per 100 cc.); and
O = extract of original wort.

10 TOTAL ACIDS.—OFFICIAL.

Proceed as directed under **XV, 23**. Express the result as lactic acid, grams per 100 cc. One cc. of N/10 sodium hydroxid is equivalent to 0.0090 gram of lactic acid.

11 VOLATILE ACIDS.—OFFICIAL.

Proceed as directed under **XV, 25**. Express the result as acetic acid, grams per 100 cc.

12 REDUCING SUGARS.—OFFICIAL.

Dilute 25 cc. of the carbon dioxid-free beer, measured at 20°C., with water at the same temperature to 100 cc. Determine the reducing sugars in 25 cc. of this solution, as directed under **VII, 41**. Express the result as grams of anhydrous maltose per 100 cc. of beer.

13 DEXTRIN.—TENTATIVE.

To 50 cc. of the carbon dioxid-free beer measured at 20°C., add 15 cc. of hydrochloric acid (sp. gr. 1.125), dilute to 200 cc., attach to a reflux condenser and keep in a boiling water bath for 2 hours. Cool, nearly neutralize with sodium hydroxid solution, complete to a volume of 250 cc., filter and determine dextrose as directed under **VII, 51** or **53**. From the number of grams of dextrose per 100 cc. of beer, subtract 1.053 times the amount of maltose as found in **12** and multiply the remainder by 0.9 to obtain the number of grams of dextrin per 100 cc. of beer.

14 DIRECT POLARIZATION.—TENTATIVE.

Read the polarization of the original sample in degrees Ventzke in a 200 mm. tube at 20°C. If the beer is turbid, clarify by shaking with alumina cream, filter and correct the reading for dilution.

15 GLYCEROL.—OFFICIAL.

Proceed as directed under **XV, 6**.

16 ASH.—OFFICIAL.

Evaporate to dryness 25 cc. of the carbon dioxid-free sample, measured at 20°C., and proceed as directed under **VII, 4**.

17 PHOSPHORIC ACID.—OFFICIAL.

To 25 cc. of the carbon dioxid-free beer, measured at 20°C., add 20 cc. of 2 per cent calcium acetate solution, evaporate to dryness and ignite at low redness to a white ash. Add 10–15 cc. of boiling nitric acid (1 to 9) and determine phosphoric acid (P_2O_5) as directed under **I, 9**.

18 PROTEIN.—OFFICIAL.

Measure, at 20°C., 25 cc. of the carbon dioxid-free beer into a Kjeldahl digestion flask, add a small amount of tannin to prevent frothing, evaporate to dryness, determine nitrogen as directed under **I**, **18**, **21** or **23**, multiply the result by 6.25 and calculate the percentage of protein.

19 PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX**.

20 COLORING MATTERS.—TENTATIVE.

Proceed as directed under **X**.

21 METALS.—TENTATIVE.

Proceed as directed under **XI**.

XVIII. VINEGARS.

(Unless otherwise noted, express results as grams per 100 cc.)

1 PHYSICAL EXAMINATION.—OFFICIAL.

Note the appearance, color, odor and taste.

2 PREPARATION OF SAMPLE.—OFFICIAL.

If the sample is turbid, filter before proceeding with the analysis.

3 SPECIFIC GRAVITY.—OFFICIAL.

Determine the specific gravity at $\frac{20° C.}{4°}$ by means of a pycnometer, as directed under XV, 3.

4 ALCOHOL.—OFFICIAL.

Measure 100 cc. of the sample into a round-bottomed distillation flask. Make faintly alkaline with saturated sodium hydroxid solution, add a small piece of paraffin, distil almost 50 cc., make up to 50 cc. at the temperature of the sample and determine the specific gravity at $\frac{20° C.}{4°}$ by means of a pycnometer. Obtain from XXX, Table 7, the per cent by volume, or grams per 100 cc., noting that the alcoholic strength of the distillate is twice that of the original vinegar.

GLYCEROL.—OFFICIAL.

5 REAGENTS.

(a) *Strong potassium dichromate solution.*—Dissolve 74.56 grams of dry, recrystallized potassium dichromate in water, add 150 cc. of concentrated sulphuric acid, cool and make up to 1 liter at 20°C. One cc. of this solution is equivalent to 0.01 gram of glycerol. The high coefficient of expansion of this strong solution makes it necessary that all volumetric measurements of the solution be made at the same temperature at which it was made up.

(b) *Dilute potassium dichromate solution.*—Measure 25 cc. of the strong potassium dichromate solution at 20°C. into a 500 cc. volumetric flask, dilute with water and make up to the mark at room temperature. Twenty cc. of this solution are equivalent to 1 cc. of (a).

(c) *Ferrous ammonium sulphate solution.*—Dissolve 30 grams of crystallized ferrous ammonium sulphate in water, add 50 cc. of concentrated sulphuric acid, cool and dilute with water to 1 liter at room temperature. One cc. of this solution is approximately equivalent to 1 cc. of (b). Its value changes slightly from day to day and it must be standardized against (b) whenever used.

(d) *Potassium ferricyanid indicator.*—Dissolve 1 gram of crystallized potassium ferricyanid in 50 cc. of water. This solution must be freshly prepared.

(e) *Milk of lime.*—Introduce 150 grams of calcium oxid, selected from clean, hard lumps, prepared preferably from marble, into a large porcelain or iron dish, slake with water, cool and add sufficient water to make 1 liter.

(f) *Silver carbonate.*—Dissolve 0.1 gram of silver sulphate in about 50 cc. of water, add an excess of sodium carbonate solution, allow the precipitate to settle and wash with water several times by decantation until the washings are practically neutral. This reagent must be freshly prepared immediately before use.

6 DETERMINATION.

All evaporations should be made on a water bath, the temperature of which is maintained at 85°–90°C.

Evaporate 100 cc. of the vinegar to 5 cc., add 20 cc. of water and again evaporate to 5 cc. to expel acetic acid. Treat the residue with about 5 grams of fine sand and 15 cc. of the milk of lime and evaporate almost to dryness, with frequent stirring, avoiding the formation of a dry crust or evaporation to complete dryness. Treat the moist residue with 5 cc. of water, rub into a homogeneous paste and then add slowly 45 cc. of absolute alcohol, washing down the sides of the dish to remove adhering paste, and stir thoroughly. Heat the mixture on a water bath, with constant stirring, to incipient boiling, transfer to a suitable vessel and centrifugalize. Decant the clear liquid into a porcelain dish and wash the residue with several small portions of hot 90 per cent alcohol by volume by aid of the centrifuge. (If a centrifuge is not available, decant the liquid through a folded filter into a porcelain dish. Wash the residue repeatedly with small portions of hot 90 per cent alcohol, twice by decantation, and then by transferring all the material to the filter. Continue the washing until the filtrate amounts to 150 cc.) Evaporate to a sirupy consistency, add 10 cc. of absolute alcohol to dissolve this residue and transfer to a 50 cc. glass-stoppered cylinder, washing the dish with successive small portions of absolute alcohol until the volume of the solution amounts to 20 cc. Then add three portions of 10 cc. each of anhydrous ether, shaking thoroughly after each addition. Let stand until clear, then pour off through a filter, and wash the cylinder and filter with a mixture of two volumes of absolute alcohol and three of anhyrous ether. If a heavy precipitate has formed in the cylinder, centrifugalize at low speed, decant the clear liquid and wash three times with 20 cc. portions of the alcohol-ether mixture, shaking the mixture thoroughly each time and separating the precipitate by means of the centrifuge. Wash the paper with the alcohol-ether mixture, and evaporate the filtrate and washings on the water bath to about 5 cc., add 20 cc. of water and again evaporate to 5 cc.; again add 20 cc. of water and evaporate to 5 cc.; finally add 10 cc. of water and evaporate to 5 cc.

These evaporations are necessary to remove all the ether and alcohol, and, when conducted at 85°–90°C., result in no loss of glycerol if the concentration of the latter is less than 50 per cent.

Transfer the residue with hot water to a 50 cc. graduated flask, cool, add the silver carbonate, prepared from 0.1 gram of silver sulphate, shake and allow to stand 10 minutes; then add 0.5 cc. of basic lead acetate solution [VII, 13 (a)], shake occasionally and allow to stand 10 minutes; make up to the mark, shake well, filter, rejecting the first portion of the filtrate, and pipette 25 cc. of the clear filtrate into a 250 cc. volumetric flask.

Add 1 cc. of concentrated sulphuric acid to precipitate the excess of lead and then 30 cc. of the strong potassium dichromate solution. Add carefully 24 cc. of concentrated sulphuric acid, rotating the flask gently to mix the contents and avoid violent ebullition, and then place in a *boiling* water bath for exactly 20 minutes. Remove the flask from the bath, dilute, cool and make up to the mark at room temperature. The amount of strong dichromate solution used must be sufficient to leave an excess of about 12.5 cc. at the end of the oxidation, the amount given above (30 cc.) being sufficient for ordinary vinegar containing about 0.35 gram or less of glycerol per 100 cc.

Standardize the ferrous ammonium sulphate solution against the dilute potassium dichromate solution by introducing from the respective burettes approximately 20 cc. of each of these solutions into a beaker containing 100 cc. of water. Complete the titration using the potassium ferricyanid solution as an outside indicator. From this titration calculate the volume (*F*) of the ferrous ammonium sulphate solution equivalent to 20 cc. of the dilute and, therefore, to 1 cc. of the strong dichromate solution.

In place of the dilute dichromate solution, substitute a burette containing the oxidized glycerol with an excess of the strong dichromate solution and ascertain how many cc. are equivalent to (*F*) cc. of the ferrous ammonium sulphate solution and, therefore, to 1 cc. of the strong dichromate solution. Then 250, divided by this last equivalent, equals the number of cc. of the strong dichromate solution present in excess in the 250 cc. flask after oxidation of the glycerol.

The number of cc. of the strong dichromate solution added, minus the excess found after oxidation, multiplied by 0.02, gives the grams of glycerol per 100 cc. of vinegar.

7 SOLIDS.—OFFICIAL.

Measure 10 cc. of the sample into a tared, flat-bottomed platinum dish of 50 mm. bottom diameter, evaporate on a boiling water bath for 30 minutes, and dry for exactly 2.5 hours in a water oven at the temperature of boiling water. Cool in a desiccator and weigh. In order that concordant results may be obtained, it is necessary to use a dish of the size and shape stated and to dry for exactly the time specified.

8 TOTAL REDUCING SUBSTANCES BEFORE INVERSION—OFFICIAL.

Proceed as directed under **VII, 25**, using 10 cc. of the sample. In the case of malt vinegar, express the results as dextrose; in all other cases, as invert sugar.

9 REDUCING SUGARS BEFORE INVERSION AFTER EVAPORATION.—OFFICIAL.

Evaporate 50 cc. of the sample on a water bath to a volume of 5 cc. Add 25 cc. of water and again evaporate to 5 cc. Transfer to a 100 cc. volumetric flask, make up to the mark, and proceed as directed under **8**, using a quantity equivalent to 10 or 20 cc. of the sample.

10 REDUCING SUGARS AFTER INVERSION.—OFFICIAL.

Proceed as directed under **9**. After the last evaporation to 5 cc. transfer to a 100 cc. volumetric flask with 70 cc. of water, and invert as directed under **VII, 14**. Nearly neutralize with sodium hydroxid solution, make up to the mark and proceed as directed under **VII, 25**, using a quantity equivalent to 10 or 20 cc. of the sample.

11 LEAD PRECIPITATE.—TENTATIVE.

To 10 cc. of the sample in a test tube, add 2 cc. of 20 per cent neutral lead acetate solution, shake and let stand 30 minutes. Describe the precipitate as turbid, light, normal, heavy or very heavy.

12 POLARIZATION.—TENTATIVE.

If the lead precipitate is normal, add to 50 cc. of the sample 5 cc. of basic lead acetate solution [**VII, 13 (a)**], shake, let stand 30 minutes, filter and polarize, preferably in a 200 mm. tube, correcting for dilution. If basic lead acetate gives only a turbidity, add to the sample, already treated with basic lead acetate, 10 cc. of alumina cream [**VII, 13** (**b**)], shake, let stand 30 minutes, filter and polarize, correcting for dilution. In the case of malt vinegar, treat 100 cc. of the sample with 5 cc. of 10 per cent phosphotungstic acid solution and filter. To 50 cc. of the filtrate add 5 cc. of the basic lead acetate solution, filter and polarize, correcting the reading obtained for dilution.

13 ASH.—OFFICIAL.

(**a**) Measure 25 cc. of the vinegar into a tared platinum dish, evaporate to dryness on a steam bath and proceed as directed under **VII, 4**.

(**b**) Evaporate 25 cc. of the sample to dryness as directed under (**a**), heat in a muffle at low heat to expel inflammable gases, treat the charred portion with a few cc. of water, and evaporate to dryness on a water bath; replace in the muffle at low redness for 15 minutes, and continue the alternate evaporation and heating until a white or gray ash is obtained, at no time exceeding a dull red heat; cool in a desiccator and weigh.

Useful information may often be obtained by noting the odor given off by the solids during charring.

14 SOLUBLE AND INSOLUBLE ASH.—OFFICIAL.

Treat the ash, obtained in **13**, as directed under **VIII, 14**.

15 ALKALINITY OF THE SOLUBLE ASH.—OFFICIAL.

Proceed as directed under **VIII, 15**, expressing the result as the number of cc. of N/10 hydrochloric acid required to neutralize the soluble ash from 100 cc. of the vinegar.

In case the relation of the ash and alkalinity of the soluble ash is abnormal, the composition of the ash should be studied, especially as to content of chlorids, sulphates, phosphates and alkalies.

16 SOLUBLE AND INSOLUBLE PHOSPHORIC ACID.—OFFICIAL.

Determine phosphoric acid in the water-soluble and water-insoluble portions of the ash as directed under **I, 9**, dissolving the water-insoluble portion in about 50 cc. of boiling nitric acid (1 to 9). Express the result as mg. of phosphorus pentoxid (P_2O_5) in 100 cc. of the vinegar.

17 TOTAL ACIDS.—OFFICIAL.

Dilute 10 cc. of the sample with recently boiled and cooled water until it appears very slightly colored, and titrate with N/2 alkali, using phenolphthalein as an indicator. One cc. of N/2 alkali is equivalent to 0.030 gram of acetic acid.

18 FIXED ACIDS.—OFFICIAL.

Measure 10 cc. of the vinegar into a 200 cc. porcelain casserole, evaporate just to dryness, add 5–10 cc. of water, and again evaporate; repeat until at least five evaporations have taken place. Add about 200 cc. of recently boiled and cooled water and titrate with N/10 alkali, using phenolphthalein as an indicator. One cc. of N/10 alkali is equivalent to 0.0067 gram of malic acid.

19 VOLATILE ACIDS.—OFFICIAL.

To obtain the volatile acids subtract the fixed acids, calculated as acetic acid, from the total acids.

20 COLOR.—OFFICIAL.

Determine the depth of color in a Lovibond tintometer by good, reflected daylight, using a ½ inch cell and the brewer's scale. Express the result in terms of a ½ inch cell.

21 FORMIC ACID[1].—OFFICIAL.

Employ the apparatus described under **IX, 40**, Fig. 4. Introduce 100 cc. of the sample into flask (*A*), add 0.4–0.5 gram of tartaric acid, and place in position as shown in Fig. 4, the flask (*B*) having previously been charged with a suspension of 15 grams of calcium carbonate in 100 cc. of water. Heat the contents of flasks (*A*) and (*B*) to boiling and distil with steam from the generator (*S*), the vapor passing first through the sample in flask (*A*), then through the boiling suspension of calcium carbonate in flask (*B*), after which it is condensed and measured in the receiver (*C*). Maintain the volume of liquid in flask (*B*) as nearly constant as possible and reduce the volume of the sample in flask (*A*) to 30–40 cc. by heating with small Bunsen flames, the distillation being continued until 1 liter of distillate is collected. Disconnect the apparatus, filter the calcium carbonate suspension, and wash the calcium carbonate that remains on the paper with a little hot water. Render the filtrate faintly acid with hydrochloric acid, add 10–15 cc. of mercuric chlorid reagent [**IX, 39** (**b**)], mix and heat on a boiling water bath for 2 hours. Filter on a tared Gooch crucible, wash the precipitate thoroughly with cold water and finally with a little alcohol. Dry in a boiling water oven for 30 minutes, cool in a desiccator, weigh and calculate the weight of formic acid present by multiplying the weight of the precipitate by 0.0975.

22 ALCOHOL PRECIPITATE.—TENTATIVE.

Evaporate 100 cc. of the vinegar to about 15 cc. When there is considerable sugar in the vinegar, if the sample is evaporated to too low a volume, a gummy or stringy precipitate is formed on adding the alcohol instead of a flocculent one. When the sugar content is high, therefore, evaporate to a volume of not less than 20 cc. To this residue add slowly and with constant stirring 200 cc. of 95 per cent alcohol by volume and allow the mixture to stand overnight. From this point proceed as directed under **XII, 18**, beginning with the sentence, "Filter and wash with 80 per cent alcohol by volume".

23 PENTOSANS.—OFFICIAL.

Proceed as directed under **VII, 63**, except that 100 cc. of the vinegar and 43 cc. of hydrochloric acid (sp. gr. 1.19) are used in beginning the distillation.

TARTARIC ACID AND TARTRATES.

24 *Qualitative Test.—Official.*

Evaporate 50 cc. of the vinegar in a porcelain dish to a volume of about 10 cc., filter into a test tube, add 1 cc. of 25 per cent calcium chlorid solution and 2 cc. of 50 per cent ammonium acetate solution and allow to stand overnight. In the presence of tartaric acid a deposit of calcium tartrate is formed, the crystals of which may be identified under the microscope by their characteristic form.

25 TOTAL TARTARIC ACID.—OFFICIAL.

Evaporate 200 cc of the sample to a sirupy consistency to remove excess of acetic acid, dilute to the original volume with water in a volumetric flask, determine the acidity as directed under **17**, and determine total tartaric acid in a 100 cc. aliquot as directed under **XV, 27**, except that 20 cc. of alcohol are used in the precipitation instead of 15 cc.

FREE MINERAL ACIDS.

26 *Logwood Method*[2].—*Tentative.*

Prepare an extract of logwood as follows: Pour 100 cc. of boiling water upon 2 grams of fresh logwood chips, allow the infusion to stand for a few hours and filter. Place drops of the liquid on a porcelain surface and dry on a water bath. Add to one of the spots a drop of the vinegar to be tested (after concentration if desirable) and evaporate to dryness. A yellow tint remains if free mineral acids are absent, a red tint if they are present.

27 *Methyl Violet Method.—Tentative.*

Add 5–10 cc. of water to 5 cc. of vinegar and, after mixing well, add 4 or 5 drops of methyl violet solution (1 part of methyl violet 2B in 10,000 parts of water). A blue or green coloration indicates the presence of a free mineral acid.

28 *Quantitative Method.—Tentative.*

To a measured amount of the sample add a measured excess of standard alkali, evaporate to dryness, incinerate and titrate the ash with standard acid, using methyl orange as an indicator. The difference between the number of cc. of alkali first added and the number of cc. of acid needed to titrate the ash represents the free mineral acid present.

29 METALS.—TENTATIVE.

Proceed as directed under **XI**.

DEXTRIN.

30 *Qualitative Test.—Tentative.*

Evaporate 100 cc. of the vinegar to a volume of about 15 cc. Add slowly and with constant stirring 200 cc. of 95 per cent alcohol by volume and allow to stand overnight. The precipitate formed should be tested for dextrin by the optical rotation and color reaction with iodin.

SPICES AND ADDED PUNGENT MATERIALS.

31 *Qualitative Test.—Tentative.*

Neutralize exactly a portion of the vinegar and test by taste and smell. Agitate the liquid with ether in a separatory funnel, remove and evaporate the ethereal layer, and note the odor and taste of the residue.

32 COLORING MATTERS.—TENTATIVE.

Proceed as directed under **X**.

33 PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX**.

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XIX. FLAVORING EXTRACTS.

VANILLA EXTRACT AND ITS SUBSTITUTES.

1 SPECIFIC GRAVITY.—OFFICIAL.

Determine the specific gravity at $\frac{20^\circ C.}{4^\circ}$ by means of a pycnometer, as directed under XV, 3.

2 ALCOHOL.—OFFICIAL.

Proceed as directed under XVI, 2 or 3.

3 GLYCEROL.—TENTATIVE.

Proceed as directed under XV, 5, 6 or 7, the method selected depending upon the amount of sugar present, employing an amount of the sample containing 0.10–0.40 gram of glycerol.

VANILLIN AND COUMARIN[1].—OFFICIAL.

(This method is not applicable to concentrated vanillin and coumarin preparations in which the amount of vanillin and coumarin present in 50 cc. exceeds the quantity dissolved by 100 cc. of water at 20°C. In such cases employ a smaller amount of the sample and dilute to 50 cc.)

4 PREPARATION OF SOLUTION.

Measure 50 cc. of the extract at 20°C. into a 250 cc. beaker with marks showing volumes of 80 and 50 cc., dilute to 80 cc. and evaporate to 50 cc. on a water bath kept at 70°C. or below. Dilute again with water to 80 cc. and evaporate to 50 cc. Transfer to a 100 cc. flask, rinsing the beaker with hot water; add 25 cc. of 8 per cent neutral lead acetate solution; make up to the mark with water, shake and allow to stand 18 hours (overnight) at 37°–40°C. Decant into a small, dry filter, reserving the filtrate for the determination of vanillin and coumarin, the lead number, 6, and the residual color, 14.

5 DETERMINATION.

Transfer a 50 cc. aliquot of the filtrate to a separatory funnel and extract with four successive 15 cc. portions of ether (previously washed twice with an equal volume of water to remove alcohol). Wash the combined ether solutions four or five times with 2 per cent ammonium hydroxid solution (2 per cent NH_3 by weight), using 10 cc. the first time and 5 cc. thereafter, and reserve the ether solution for the determination of coumarin. Slightly acidify the combined ammoniacal solutions with hydrochloric acid; cool and extract in a separatory funnel with four portions of washed ether, using about 40 cc. altogether. Evaporate the ethereal solutions at room temperature, dry over sulphuric acid and weigh. If the residue is considerably discolored or gummy, re-extract in the dry state with boiling petroleum ether (b. p. 40°C. or below) not less than 15 times; evaporate the solvent, dry and weigh. The residue should now be white, crystalline vanillin, with a melting point of approximately 80°C. A small amount of this residue, dissolved in 2 drops of concentrated hydrochloric acid, should develop a pink color upon the addition of a crystal of resorcin.

Evaporate at room temperature the original ether extract of the sample, from which the vanillin has been removed by means of ammonium hydroxid, and dry over sulphuric acid. The residue, if pure coumarin, should melt at approximately 67°C. and should respond to Leach's test for coumarin as follows: A small portion of the residue, dissolved in not more than 0.5 cc. of hot water, should yield a brown precipitate upon the addition of a few drops of N/10 iodin. This precipitate finally gathers in green flecks, leaving a clear, brown solution. The reaction is especially marked if the reagent is applied with a glass rod to a few drops of the solution on a white plate or tile.

6 LEAD NUMBER[2].—OFFICIAL.

To a 10 cc. aliquot of the filtrate from the lead acetate precipitate, as obtained in **4**, add 25 cc. of water, 0.5–1.0 cc. of sulphuric acid, and 100 cc. of 95 per cent alcohol by volume. Let stand overnight, filter on a Gooch crucible, wash with 95 per cent alcohol, dry at a moderate heat, ignite at low redness for 3 minutes, taking care to avoid the reducing flame, and weigh. Conduct a blank determination employing water containing 4 or 5 drops of glacial acetic acid in place of the sample. The lead number is calculated by the following formula:

$$P = \frac{100 \times 0.6831\ (S - W)}{5} = 13.66\ (S - W) \text{ in which}$$

P = lead number (grams of metallic lead in the precipitate obtained from 100 cc. of the sample);
S = grams of lead sulphate corresponding to 2.5 cc. of the lead acetate solution as determined in a blank analysis; and
W = grams of lead sulphate obtained in 10 cc. of the filtrate from the lead acetate precipitate, as obtained in **4**.

7 TOTAL SOLIDS.—OFFICIAL.

Proceed as directed under **VIII, 4**, employing 10 cc. of the sample.

8 ASH.—OFFICIAL.

Evaporate 10 cc. of the extract and determine the ash as directed under **VII, 4**.

9 ASH CONSTITUENTS.

Proceed as directed under **II** or **XXVII, 21–26**, inclusive.

10 SUCROSE.—OFFICIAL.

Determine as directed under **VII, 14** or **18**.

VANILLA RESINS.

11 *Qualitative Test.—Tentative.*

Place 50 cc. of the extract in a glass dish and evaporate the alcohol on a water bath. When the alcohol is removed, make up to about the original volume with hot water. If alkali has not been used in the manufacture of the extract, the resins will appear as a flocculent red to brown residue. Acidify with acetic acid to free the resins from the bases, separating the resins completely and leaving a partly decolorized, clear, supernatant liquid after standing a short time. Collect the resins on a filter, wash with water and reserve the filtrate for further tests.

Place a portion of the filter with the attached resins in a few cc. of dilute potassium hydroxid solution. The resins are dissolved, giving a deep red solution; acidify, and the resins are precipitated.

Dissolve a portion of the resins in alcohol. To one portion add a few drops of ferric chlorid solution; to another portion, hydrochloric acid; neither produces any marked change in color. Most resins, however, in alcoholic solution give color reactions with ferric chlorid or hydrochloric acid.

To a portion of the filtrate obtained above add a few drops of basic lead acetate solution. The precipitate is so bulky as to almost solidify, due to the excessive amount of organic acids, gums and other extractive matter. The filtrate from this precipitate is almost colorless.

Test another portion of the filtrate from the resin for tannin with a solution of gelatin. Tannin is present in varying but small quantities, but should not be present in great excess.

12 METHYL ALCOHOL.—OFFICIAL.

Proceed as directed under **XVI**, **16**, **17** or **18**, using the distillate from the determination of alcohol, **2**.

13 COLOR VALUE.—TENTATIVE.

Pipette 2 cc. of the extract into a 50 cc. graduated flask and make up to the mark with a mixture of equal parts of 95 per cent alcohol by volume and water. Determine the color value of this diluted extract in terms of red and yellow by means of a Lovibond tintometer, using a 1 inch cell. To obtain the color value of the original extract, multiply the figures for each color by 25.

14 RESIDUAL COLOR AFTER PRECIPITATION WITH LEAD ACETATE[3].—TENTATIVE.

Determine the color value, in terms of red and yellow, of the filtrate from the lead acetate precipitate as obtained in **4**, using a 1 inch Lovibond cell. Multiply the reading by 2 to reduce the results to the basis of the original extract. If the actual reading of the solution is greater than 5 red and 15 yellow, as may happen if the extract is highly colored with caramel, a ½ or ¼ inch cell should be employed, and the readings multiplied, respectively, by 4 or 8. Divide the figures for red and yellow, respectively, by the corresponding figures of the original extract and multiply the quotients by 100, to obtain the percentages of the two colors remaining in the lead acetate filtrate.

Calculate also the ratio of red to yellow in both extract and lead acetate filtrate.

15 COLORS INSOLUBLE IN AMYL ALCOHOL.—TENTATIVE.

Proceed as directed under **XVI**, **22**, using 25 cc. of the extract and shaking with 25 cc. of the Marsh reagent instead of 20 cc.

16 COLORING MATTERS OTHER THAN CARAMEL.—TENTATIVE.

Proceed as directed under **X**.

LEMON AND ORANGE EXTRACTS.

17 SPECIFIC GRAVITY.—OFFICIAL.

Determine the specific gravity at $\frac{20^\circ \text{C.}}{4^\circ}$ by means of a pycnometer, as directed under **XV**, **3**.

18 ALCOHOL.—OFFICIAL.

Dilute 50 cc. of the extract, measured at 20°C., with water to about 200 cc., allow the mixture to stand until the oil separates in a clear layer at the top, or centrifugalize, then make up to the mark, using the lower meniscus of the oil. Pour the mixture into a dry Erlenmeyer flask containing 5 grams of light magnesium carbonate, stopper, shake well and filter quickly through a large, dry, folded filter. Introduce a 150 cc. aliquot of the filtrate, measured at 20°C., into a 300–500 cc. distillation flask, attach the flask to a vertical condenser and distil almost 100 cc. Complete the volume of the distillate to 100 cc. at 20°C., mix well and determine the specific gravity at $\frac{20°C.}{4°}$ Ascertain the corresponding per cent of alcohol by volume from **XXX**, Table 7, and multiply the result thus obtained by $2\frac{2}{3}$ to obtain the precentage of alcohol by volume in the original sample.

19 GLYCEROL.—TENTATIVE.

Proceed as directed under **3**.

LEMON AND ORANGE OILS.

20 *By Polarization.—Official.*

Without diluting polarize the extract at 20°C. in a 200 mm. tube. Divide the reading in degrees Ventzke by 3.2 in the case of lemon extract and by 5.2 in the case of orange extract; in the absence of other optically active substances, the result will be the percentage of oil by volume. A small amount of cane sugar is occasionally present; if so, determine as directed under **28** and correct the reading accordingly.

21 *By Precipitation.—Official.*

Pipette 20 cc. of the extract into a Babcock milk bottle, add 1 cc. of hydrochloric acid (1 to 1), then 25–28 cc. of water previously warmed to 60°C., mix, let stand in water at 60°C. for 5 minutes, centrifugalize for 5 minutes, fill with warm water to bring the oil into the graduated neck of the flask, again centrifugalize for 2 minutes, place the flask in water at 60°C. for a few minutes and note the per cent of oil by volume. If oil of lemon is present in amounts over 2 per cent, add 0.4 per cent to the percentage of oil noted to correct for the solubility of the oil. If less than 2 per cent and more than 1 per cent is present, add 0.3 per cent for this correction.

When the extract is made in accordance with the U. S. P., the results by the methods given under **20** and **21** should agree within 0.2 per cent.

To obtain the per cent by weight from the per cent by volume, as found by either of these methods, multiply the volume percentage by 0.86 in the case of lemon extracts, and by 0.85 in the case of orange extracts, and divide the result by the specific gravity of the original extract.

TOTAL ALDEHYDES[4].—OFFICIAL.

22 REAGENTS.

(**a**) *Aldehyde-free alcohol.*—Allow 95 per cent alcohol by volume, containing 5 grams of meta-phenylendiamin hydrochlorid per liter, to stand for at least 24 hours with frequent shaking. (Nothing is gained by previous treatment with potassium hydroxid.) Boil under a reflux condenser for at least 8 hours, longer if necessary, allow to stand overnight and distil, rejecting the first 10 and the last 5 per cent which come over. Store in a dark, cool place in well filled bottles. Twenty-five cc. of this alcohol, on standing 20 minutes at 14°–16°C. with 20 cc. of the sulphite-fuchsin solution, should develop only a faint pink coloration. If a stronger color is developed, repeat the treatment with meta-phenylendiamin hydrochlorid as above.

(**b**) *Sulphite-fuchsin solution.*—Dissolve 0.5 gram of fuchsin in 250 cc. of water, add an aqueous solution of sulphur dioxid containing 16 grams of the gas, allow to stand until colorless or nearly so and make up to 1 liter with water. Let stand 12 hours before using and keep in a refrigerator. This solution is liable to deteriorate and should be reasonably fresh when used.

(c) *Standard citral solution.*—Use 0.5 or 1 mg. of C. P. citral per cc. in 50 per cent aldehyde-free alcohol.

23 DETERMINATION.

Weigh approximately 25 grams of the extract in a stoppered weighing flask, transfer to a 50 cc. flask and make up to the mark at room temperature with aldehyde-free alcohol. Measure, at room temperature, 2 cc. (or other suitable amount) of this solution into a comparison tube. Add 25 cc. of the aldehyde-free alcohol (previously cooled to 14°–16°C.), then 20 cc. of the sulphite-fuchsin solution (also cooled) and finally make up to the 50 cc. mark with aldehyde-free alcohol. Mix thoroughly, stopper and keep at 14°–16°C. for 15 minutes. Prepare a standard for comparison at the same time and in the same manner, using 2 cc. of the standard citral solution, and compare the colors developed. Calculate the amount of citral present and repeat the determination, using a quantity sufficient to give the sample approximately the strength of the standard. From this result calculate the amount of citral in the sample. If the comparisons are made in Nessler tubes, standards containing 1, 1.5, 2, 2.5, 3, 3.5, and 4 mg. of citral, may be prepared and the trial comparison made against these, the final comparison being made with standards lying between 1.5 and 2.5 mg. with 0.25 mg. increments.

It is absolutely essential to keep the reagents and comparison tubes at the required temperature, 14°–16°C. Where the comparisons are made in a bath (this being possible only where the bath is of glass), the standards should be discarded within 25 minutes after adding the sulphite-fuchsin solution. Give samples and standards identical treatment.

CITRAL[5].—OFFICIAL.

24 REAGENTS.

(a) *Meta-phenylendiamin hydrochlorid solution.*—Prepare a 1 per cent solution of meta-phenylendiamin hydrochlorid in 95 per cent alcohol by volume. Decolorize, if necessary, by shaking with fuller's earth and filter through a double filter. The solution should be bright and clear, free from suspended matter, and practically colorless. Prepare this solution only for immediate use.

(b) *Alcohol.*—For the analysis of lemon extracts, 90–95 per cent alcohol by volume should be used, but for terpeneless extracts, 40–50 per cent alcohol by volume is sufficient. Filter to remove any suspended matter. The alcohol need not be purified from aldehyde. If not practically colorless, render slightly alkaline with sodium hydroxid and distil.

25 DETERMINATION.

All of the operations may be carried on at room temperature. Weigh 25 grams of the extract into a 50 cc. graduated flask and make up to the mark with alcohol. Stopper the flask and mix the contents thoroughly. Pipette 2 cc. (or other suitable amount) of this solution into a colorimeter tube; add 10 cc. of the meta-phenylendiamin hydrochlorid solution and complete the volume to 50 cc. (or other standard volume) with alcohol. Compare at once the color with that of the standard, prepared at the same time, using 2 cc. of standard citral solution and 10 cc. of the meta-phenylendiamin hydrochlorid solution, and making up to standard volume with alcohol. From the result of this first determination, calculate the amount of standard citral solution that should be used in order to give approximately the same citral strength as the sample under examination; then repeat the determination.

26 TOTAL SOLIDS.—OFFICIAL.

Proceed as directed under **XVI, 5**, employing 10 cc. of the sample measured at 20°C.

27 ASH.—OFFICIAL.

Ignite the residue from 10 cc. of the extract as directed under **VII, 4**.

28 SUCROSE.—OFFICIAL.

Neutralize the normal weight of the extract, evaporate to dryness, wash several times with ether, dissolve in water and determine as directed under **VII, 14** or **18**.

29 METHYL ALCOHOL.—OFFICIAL.

Proceed as directed under **XVI, 16, 17** or **18**, using the distillate from the determination of alcohol, **18**.

COLORING MATTERS.

30 GENERAL.—TENTATIVE.

Proceed as directed under **X**.

31 LEMON AND ORANGE PEEL COLOR.—TENTATIVE.

Place a few cc. of the extract in each of two test tubes; to one, add slowly 3-4 volumes of concentrated hydrochloric acid; to the other, several drops of concentrated ammonium hydroxid. If the color is due to lemon or orange peel only, it is materially deepened by such treatment.

LEMON AND ORANGE OILS.

32 SPECIFIC GRAVITY.—OFFICIAL.

Determine the specific gravity at $\frac{20^\circ \text{C.}}{4^\circ}$ by means of a pycnometer, as directed under **XV, 3**.

33 INDEX OF REFRACTION.—OFFICIAL.

Determine the index of refraction with any standard instrument, making the reading at 20°C.

34 OPTICAL ROTATION.—OFFICIAL.

Determine the rotation at 20°C. with any standard instrument, using a 50 mm. tube and sodium light. The results should be stated in angular degrees on a 100 mm. basis. If instruments having the sugar scale are used, the reading on orange oils is above the range of the scale, but readings may be obtained by the use of standard laevo rotatory quartz plates, or by the use of a 25 mm. tube. The true rotation can not be obtained by diluting the oil with alcohol and correcting the rotation in proportion to the dilution.

CITRAL.

Kleber Method[6].—*Official.*

35 REAGENTS.

(a) *Phenylhydrazin solution.*—Prepare a 10 per cent solution in absolute alcohol. A sufficiently pure product can be obtained by distilling the commercial article in vacuo, rejecting the first portions coming over which contain ammonia.

(b) *N/2 hydrochloric acid.*

36 DETERMINATION.

Weigh accurately about 15 grams of the sample into a small, glass-stoppered flask; add 10 cc. of the phenylhydrazin solution. Allow to stand 30 minutes at room temperature, titrate with N/2 hydrochloric acid, using either methyl or ethyl orange as an indicator. Titrate similarly 10 cc. of the phenylhydrazin solution. The difference in the number of cc. of N/2 acid used in these two titrations, multiplied by the factor 0.076, gives the weight of citral in the sample. If difficulty is experienced in detecting the end point of the reaction, titrate until the solution is distinctly acid, transfer to a separatory funnel, and draw off the alcoholic portion. Wash the oil with water, adding the washings to the alcoholic solution, titrate back with N/2 alkali and make the necessary corrections.

37 *Hiltner Method*[6].—*Official.*

Weigh 2 grams of lemon oil or 8 grams of orange oil into a 100 cc. graduated flask, dilute to the mark with 95 per cent alcohol by volume and proceed as directed under **25**, using 2 cc. of the dilute solution for the comparison.

38 TOTAL ALDEHYDES[4].—OFFICIAL.

Weigh a small quantity of the sample into a small, stoppered flask and dilute with aldehyde-free alcohol in the proportion of 2 grams of lemon oil or 4 grams of orange oil to 100 cc. of solution. Determine the total aldehydes as directed under **23**, expressing the result as citral.

39 PHYSICAL CONSTANTS OF THE 10 PER CENT DISTILLATE[7].—OFFICIAL.

Place 50 cc. of the sample in a 3-bulb Ladenburg flask having the main bulb 6 cm. in diameter and of 120 cc. capacity and the condensing bulbs of the following dimensions: 3.5 cm., 3 cm., 2.5 cm.; the distance from the bottom of the flask to the opening of the side arm should be 20 cm. Distil the oil at the rate of 2 cc. per minute until 5 cc. have been distilled. Determine the refractive index and rotation of this distillate as directed under **33** and **34**.

40 PINENE[8].—OFFICIAL.

Mix the 10 per cent distillate, obtained in **39**, with 5 cc. of glacial acetic acid; cool the mixture thoroughly in a freezing bath and add 10 cc. of ethyl nitrite. Then add slowly, with constant stirring, 2 cc. of hydrochloric acid (2 to 1). Keep the mixture in the freezing bath 15 minutes. Filter off the crystals formed, using suction, and wash with 95 per cent alcohol by volume. Return the combined filtrate and washings to the freezing bath for 15 minutes. Filter off the crystals formed, using the original filter paper. Wash the combined crops of crystals thoroughly with alcohol. Dry at room temperature and dissolve in a minimum amount of chloroform. Add methyl alcohol to the chloroform solution, a little at a time, until the nitroso-chlorids crystallize out, mount the separated and dried crystals in olive oil and examine under the microscope. Pinene nitroso-chlorid crystals have irregular pyramidal ends while limonene nitroso-chlorid crystallizes in needles.

ALMOND EXTRACT.

41 ALCOHOL.—TENTATIVE.

Inasmuch as almond extract usually contains only about 1 per cent of almond oil, the alcohol can, in most cases, be calculated from the specific gravity of the extract. If the extract is high in solids, determine the alcohol as follows: Add 25 cc. of the extract, measured at 20°C., to 75 cc. of saturated sodium chlorid solution in a separatory funnel and extract twice with 50 cc. portions of petroleum ether (b. p. 40°–60°C.). Collect the petroleum ether extract in a second separatory funnel and wash twice with 2 portions (25 cc.) of saturated brine. Combine the original salt solution with the washings; add a little powdered pumice and distil into a 100 cc. flask. When almost 100 cc. have been distilled, make up to the mark with water at 20°C. and determine alcohol from the specific gravity, as directed under **XVI, 4**.

BENZALDEHYDE.—TENTATIVE.

42 REAGENT.

Phenylhydrazin solution.—Add 3 cc. of glacial acetic acid to 40 cc. of water and mix with 2 cc. of phenylhydrazin.

43 DETERMINATION.

Measure out two portions of 10 cc. each of the extract into 300 cc. Erlenmeyer flasks and add 10 cc. of the phenylhydrazin solution to one flask and 15 cc. to the other. Allow to stand overnight in a dark place, add 200 cc. of water and filter on a tared Gooch crucible, provided with a thin layer of asbestos. Wash first with cold water, finally with 10 cc. of 10 per cent alcohol, and dry for 3 hours in a vacuum oven at 70°C., or to constant weight over sulphuric acid. The weight of the precipitate, multiplied by the factor 5.408, gives the weight of benzaldehyde in 100 cc. of the sample. If duplicate determinations do not agree, repeat the operation, using a larger quantity of the phenylhydrazin solution.

44 BENZOIC ACID[9].—TENTATIVE.

Measure 10 cc. of the extract into a 100 cc. flask, add 10 cc. of a 10 per cent sodium hydroxid solution and 20 cc. of hydrogen peroxid solution (U. S. P.); cover with a watch glass and place in a water oven. Oxidation of the aldehyde to benzoic acid begins almost immediately and should be continued 5–10 minutes after all odor of benzaldehyde has disappeared, which usually requires 20–30 minutes. Remove the flask from the water oven, transfer the contents to a separatory funnel, rinsing off the watch glass, add 10 cc. of dilute sulphuric acid solution (1 to 5) and cool the contents of the funnel to room temperature under the water tap. Extract the benzoic acid with 4 portions of 25, 25, 20, and 20 cc. of ether, respectively, and wash the combined extracts with two portions of 5–10 cc. of water, or until all sulphuric acid is removed. Filter into a tared dish, evaporate at room temperature, dry overnight in a desiccator and weigh the benzoic acid. Multiply the result by 10.

Multiply the grams per 100 cc. of benzaldehyde obtained in **43** by 1.151 to obtain the equivalent of benzoic acid, and subtract this from the grams per 100 cc. of total benzoic acid obtained above. The difference is the grams of benzoic acid per 100 cc. of the extract.

HYDROCYANIC ACID.

45 *Qualitative Test.—Tentative.*

Add several drops of ferrous sulphate solution and a single drop of ferric chlorid solution to several cc. of the extract. Mix thoroughly, add sodium hydroxid solution, drop by drop, until no further precipitate forms and then dilute hydrochloric acid to dissolve the precipitate. In the presence even of small amounts of hydrocyanic acid, a Prussian blue coloration or suspension will develop.

46 *Quantitative Method.—Tentative.*

(In the absence of chlorids.)

Measure 25 cc. of the extract into a small flask and add 5 cc. of freshly precipitated magnesium hydroxid (chlorin-free). Titrate with N/10 silver nitrate solution, using potassium chromate as an indicator; 1 cc. of N/10 silver nitrate is equivalent to 0.00268 gram of hydrocyanic acid.

NITROBENZOL.

47 *Qualitative Test.—Tentative.*

Boil a few cc. of the extract with some zinc dust and acetic acid and filter. Add to the filtrate a drop of chloroform, make strongly alkaline with sodium hydroxid solution and heat. The presence of nitrobenzol in the original extract is indicated by the development of the characteristic odor of phenylisonitrile.

CASSIA, CINNAMON AND CLOVE EXTRACTS.

48 ALCOHOL.—TENTATIVE.

Determine as directed under **41**.

49 OIL[9].—TENTATIVE.

Transfer 10 cc. of the extract to a separatory funnel, add 30 cc. of water, acidify with 1 cc. of hydrochloric acid (1 to 1) and extract three times with ether, using not less than 100 cc. altogether. Wash the combined ether solutions twice with water and, in the case of cinnamon extract, dry by shaking with a small amount of granulated calcium chlorid. Transfer to a tared, wide-mouthed weighing bottle and evaporate the ether as rapidly as possible on a boiling water bath, rotating the liquid upon the sides of the bottle in order to rid the residual oil of traces of ether. Weigh the residue and divide the weight by the specific gravity of the oil in order to obtain the per cent of oil by volume. In the case of clove oil, allow the weighing bottle to remain in the balance case until the usual film of moisture has evaporated. The time of weighing, however, should not be delayed over 3 minutes.

Determine the refractive index of the residual oils at 20°C.

Dissolve a drop of the oil in several drops of alcohol and add a drop of ferric chlorid solution. The following tabulation gives the specific gravity, refractive index at 20°C. and color reaction with ferric chlorid solution:

OIL	SPECIFIC GRAVITY	REFRACTIVE INDEX AT 20°C.	COLOR REACTION WITH FERRIC CHLORID SOLUTION
Cassia	1.05	1.585–1.600	Brown
Cinnamon	1.03	1.590–1.599	Green
Cloves	1.055	1.560–1.565	Deep blue

GINGER EXTRACT.

50 ALCOHOL.—TENTATIVE.

Determine as directed under **XVI, 4.**

51 SOLIDS.—TENTATIVE.

Evaporate 10 cc. of the extract nearly to dryness on a water bath, dry for 2 hours in a water oven and weigh.

52 GINGER.—TENTATIVE.

Dilute 10 cc. of the extract to 30 cc., evaporate to 20 cc., decant into a separatory funnel and extract with an equal volume of ether. Allow the ether to evaporate spontaneously in a porcelain dish, and to the residue add 5 cc. of 75 per cent sulphuric acid and about 5 mg. of vanillin. Allow to stand 15 minutes and add an equal volume of water; in the presence of ginger extract an azure blue color develops.

53 CAPSICUM.—TENTATIVE.

To 10 cc. of the extract add cautiously dilute sodium hydroxid solution until the solution reacts very slightly alkaline with litmus paper. Evaporate at about 70°C. to approximately one-fourth the original volume and render slightly acid with dilute sulphuric acid, testing with litmus paper. Transfer to a separatory funnel, rinsing the dish with water, and extract with an equal volume of ether, avoiding the formation of an emulsion by shaking the funnel gently 1–2 minutes. Draw off the lower layer and wash the ether extract once with about 10 cc. of water. Transfer the washed ether extract to a small evaporating dish, render decidedly alkaline with N/2 alcoholic potassium hydroxid and evaporate at about 70°C. until the residue is pasty; then add about 20 cc. more of N/2 alcoholic potash and allow to stand on a steam bath until the gingerol is completely saponified (about 30 minutes). Dissolve the residue in a little water and transfer with water to a small separatory funnel. The volume should not exceed 50 cc. Extract the alkaline solution with an equal volume of ether. Wash the ether extract repeatedly with small amounts of water until no longer alkaline to litmus. Transfer the washed extract to a small evaporating dish, and allow the ether to evaporate spontaneously. Finally test the residue for capsicum by moistening the tip of the finger, rubbing it on the bottom and sides of the dish and then applying the finger to the end of the tongue. A hot, stinging or prickly sensation, which persists for several minutes, indicates capsicum or other foreign pungent substances.

PEPPERMINT, SPEARMINT AND WINTERGREEN EXTRACTS.

54 ALCOHOL.—TENTATIVE.

Proceed as directed under **41.**

55 OIL[10].—TENTATIVE.

Pipette 10 cc. of the extract into a Babcock milk bottle, add 1 cc. of carbon disulphid, mix thoroughly, then add 25 cc. of cold water and 1 cc. of concentrated hydrochloric acid. Close the mouth of the bottle and shake vigorously; centrifugalize for 6 minutes and remove all but 3–4 cc. of the supernatant liquid, which should be practically clear, by aspirating through a glass tube of small bore. Connect the stem of the bottle with a filter pump, immerse the bottle in water kept at approximately 70°C. for 3 minutes, remove from the bath every 15 seconds and shake vigorously. Continue in the same manner for 45 seconds, using a boiling water bath. Remove from the bath and shake while cooling. Disconnect from the suction and fill the bottle to the neck with saturated salt solution at room temperature, centrifugalize for 2 minutes and read the volume of the separated oil from the top of the meniscus. Multiply the reading by 2 to obtain the per cent of oil by volume. In the case of wintergreen, use as a floating medium a mixture of 1 volume of concentrated sulphuric acid and 3 of saturated sodium sulphate solution.

56 METHYL SALICYLATE IN WINTERGREEN EXTRACT[9].—TENTATIVE.

Mix 10 cc. of the extract with 10 cc. of 10 per cent potassium hydroxid solution. Heat on a steam bath until the volume is reduced about one-half, add a distinct excess of hydrochloric acid (1 to 1), cool and extract with three portions of ether, 40, 30 and 20 cc., respectively. Filter the extract through a dry filter into a weighed dish, wash the paper with 10 cc. of ether and allow the filtrate and washings to evaporate spontaneously. Dry in a desiccator containing sulphuric acid and weigh. Multiply the weight of salicylic acid so found by 9.33 to obtain the per cent by volume of methyl salicylate in the sample.

ANISE AND NUTMEG EXTRACTS.

57 OIL[9].—TENTATIVE.

To 10 cc. of the extract in a Babcock milk bottle add 1 cc. of hydrochloric acid (1 to 1), then sufficient half saturated salt solution, previously heated to 60°C., to fill the flask nearly to the neck. Cork and let stand in water at 60°C. for about 15 minutes, rotate occasionally and centrifugalize for 10 minutes at about 800 revolutions per minute. Add brine till the oil rises into the neck of the bottle, and again centrifugalize for 10 minutes. If the separation is not satisfactory, or the liquid is not clear, cool to about 10°C. and centrifugalize for an additional 10 minutes. Multiply the reading by 2 to obtain the percentage of oil by volume.

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XX. MEAT AND MEAT PRODUCTS.

MEAT.

1 PREPARATION OF SAMPLE.—OFFICIAL.

In the case of fresh meat, separate the sample as completely as possible from the bones and pass through a sausage mill rapidly and repeatedly until thoroughly mixed and macerated. Chill the sample to prevent decomposition and begin all determinations as soon as practicable after the sample is prepared.

In the case of canned meats, pass the entire contents of a can through a sausage mill as directed above. Remove sausage from the casings and mix by repeated grinding in a sausage mill. Dry the portion of the sample, which is not needed for analysis, either in vacuo or by evaporating with alcohol, extract the fat with gasoline (b. p. below 60°C.), allow the gasoline to evaporate spontaneously and expel the last traces by heating for a short time on a steam bath. Do not heat the meat or separated fat longer than necessary, owing to their tendency to decompose. Reserve the fat for examination according to the methods given under **XXII**. Keep the fat in a cool place and complete the examination before it becomes rancid.

2 MOISTURE.—OFFICIAL.

Proceed as directed under **VII, 2** or **3**, using the latter method in cases in which it is desired to employ the dried sample for further determinations.

3 ASH.—OFFICIAL.

Proceed as directed under **VII, 4**.

4 CRUDE FAT OR ETHER EXTRACT.—OFFICIAL

Proceed as directed under **VII, 10**.

5 TOTAL PHOSPHORUS.—OFFICIAL.

Destroy the organic matter as directed under **I, 5 (a)**, **(b)**, **(c)** or **(d)** and proceed as directed under **I, 6** or **9**.

6 TOTAL NITROGEN.—OFFICIAL.

Proceed as directed under **I, 18, 21** or **23**, using about 2 grams of the fresh sample. In the Kjeldahl and Gunning methods digest with sulphuric acid for at least 4 hours; in the Kjeldahl-Gunning-Arnold method, for 2 hours after the mixture has become clear.

AMMONIA.

Aeration Method[1].—*Tentative.*

7 APPARATUS.

Employ the apparatus illustrated in Fig. 7; *A* is a wash bottle one-fourth full of 10 per cent sulphuric acid; *B* is a tube containing the sample; *C* is a rubber disc and *D* is a 5 cc. bulb to prevent spray from being carried over into the tube (*E*) which contains the standard acid; *F* is a safety bottle.

8 DETERMINATION.

Introduce 2-4 grams of the finely divided meat into the tube (*B*) and add 20 cc. of ammonia-free water. Place a measured amount of N/25 or N/50 sulphuric or hydrochloric acid in tube (*E*). Then add 1 cc. of saturated potassium oxalate solution to the sample in tube (*B*), introduce a few drops of kerosene and finally add just sufficient saturated potassium carbonate solution to render the mixture alkaline. Place the tubes in position at once, pass air through the apparatus and titrate the standard acid in tube (*E*) at hourly intervals, until ammonia ceases to be given off, using methyl red, cochineal or congo red as an indicator. If preferred, the ammonia collected in tube (*E*) may also be determined by nesslerizing as directed under **III, 11.**

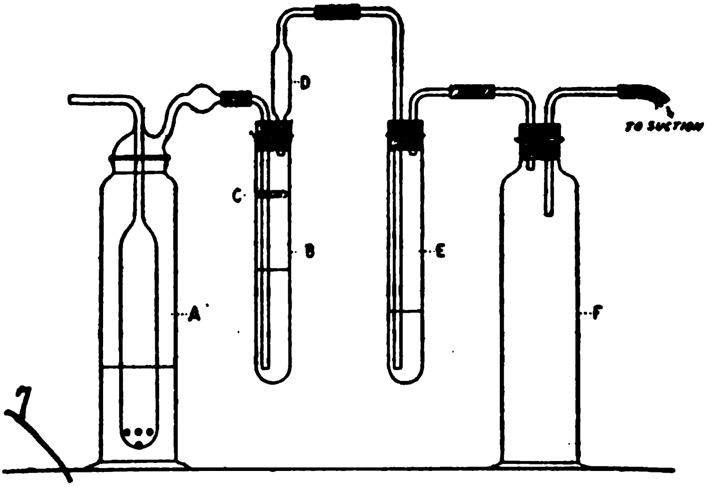

FIG. 7. APPARATUS FOR AMMONIA DETERMINATION.

NITRATES.

Ferrous Chlorid Method[2].—*Tentative.*

9 REAGENT.

Ferrous chlorid solution.—Dissolve nails or other small pieces of iron in concentrated hydrochloric acid, keeping an excess of iron present until the evolution of gas ceases. Keep the solution in 50 cc. glass-stoppered bottles entirely filled. Employ only freshly opened bottles of the reagent for the determination.

10 APPARATUS.

Provide a 250 cc. flask with a 2-holed rubber stopper. Through one of the holes pass the stem of a funnel having a glass stop-cock, and into the other fit a delivery tube leading downward at an angle from the flask to a trough containing water. Terminate the upper end of the delivery tube just below the rubber stopper in the flask and place the lower end under the surface of the water in the trough, the exit being immediately beneath the mouth of an inverted measuring tube, filled with 40 per cent potassium hydroxid solution. Cover the trough end of the delivery tube with a piece of rubber tubing to prevent fracture, but arrange the latter so that it will not interfere with the free exit of gas. On the delivery tube midway between the flask and the measuring tube place a short length of rubber tubing and a pinch-cock.

11 DETERMINATION.

Extract 100 grams of finely ground meat by boiling repeatedly with successive small portions of water, decanting the extracts through a muslin or paper filter into a casserole, and concentrate the combined extracts to a volume of about 50 cc.

Introduce 50 cc. of the ferrous chlorid solution and 50 cc. of 10 per cent hydrochloric acid into the flask, close the stop-cock of the funnel, open the pinch-cock on the delivery tube, move the end of the latter so that escaping air will not pass into the measuring tube, and boil the contents of the flask until the air is expelled, as indicated by a slight pressure against the fingers when the rubber tubing is compressed after momentary removal of the flame. Now close the delivery tube with the pinch-cock and place the exit end beneath the measuring tube. Introduce the concentrated extract of the sample into the flask, a little at a time, through the funnel tube, opening the pinch-cock on the delivery tube and boiling the contents of the flask at intervals to force the nitric oxid gas into the measuring tube. Finally rinse the casserole and the funnel tube with a little boiled water, add the rinsings to the contents of the evolution flask in the manner just described and boil until nitric oxid no longer passes over into the measuring tube. Note the volume of nitric oxid contained in the tube, the temperature, and barometric pressure and calculate the volume of nitric oxid at 0°C. and 760 mm. pressure. One cc. of nitric oxid at 0°C. and 760 mm. pressure is equivalent to 0.004512 gram of potassium nitrate.

Phenoldisulphonic Acid Method[3].—*Tentative.*

12 REAGENTS.

(a) *Phenoldisulphonic acid solution.*—Heat 6 grams of phenol with 37 cc. of concentrated sulphuric acid on a water bath, cool and add 3 cc. of water.

(b) *Standard comparison solution.*—Dissolve 1 gram of pure, dry potassium nitrate in water and dilute to 1 liter. Evaporate 10 cc. of this solution to dryness on a steam bath, add 2 cc. of the phenoldisulphonic acid solution, mix quickly and thoroughly by means of a glass rod, heat for about a minute in a steam bath and dilute to 100 cc. One cc. of this solution is equivalent to 0.1 mg. of potassium nitrate. Prepare a series of standard comparsion tubes by introducing amounts ranging from 1 to 20 cc. of this solution (0.1–2.0 mg. of potassium nitrate) into 50 cc. Nessler tubes, adding 5 cc. of strong ammonium hydroxid to each and diluting to 50 cc. These standard tubes are permanent for several weeks if kept tightly stoppered.

13 DETERMINATION.

Weigh 1 gram of the sample into a 100 cc. flask, add 20–30 cc. of water and heat on a steam bath for 15 minutes, shaking occasionally. Add 3 cc. of saturated silver sulphate solution for each per cent of sodium chlorid present, then 10 cc. of basic lead acetate solution and 5 cc. of alumina cream, shaking after each addition. Make up to the mark with water, shake and filter through a folded filter, returning the filtrate to the filter until it runs through clear. Evaporate 25 cc. of the filtrate to dryness, add 1 cc. of the phenoldisulphonic acid solution, mix quickly and thoroughly by means of a glass rod, add 1 cc. of water and 3 or 4 drops of concentrated sulphuric acid and heat on a steam bath for 2–3 minutes, being careful not to char the material. Then add about 25 cc. of water and an excess of ammonium hydroxid, transfer to a 100 cc. graduated flask, add 1–2 cc. of alumina cream if not perfectly clear, dilute to the mark with water and filter. Fill a 50 cc. Nessler tube to the mark with the filtrate and determine the amount of potassium nitrate present in the sample by comparison with the standard comparison tubes. If the solution is too dark for comparison with the standards, dilute with water and correct the result accordingly.

STARCH.

(In chopped meat, sausage, deviled meat, etc.)

14 *Qualitative Test.—Tentative.*

Treat 5–6 grams of the sample with boiling water for 2–3 minutes, cool the mixture and test the supernatant liquid with iodin solution. In using this test, a small amount of starch may be present as the result of the use of spices. If a marked reaction is given, however, it may be concluded that starch or flour has been added, and a quantitative determination may be made. The qualitative method may be replaced by a microscopic examination, which discloses not only the presence of added starch, but also the variety employed.

15 *Quantitative Method[4].—Tentative.*

Treat in a 200 cc. beaker 10 grams of the finely divided sample with 75 cc. of an 8 per cent solution of potassium hydroxid in 95 per cent alcohol by volume and heat on a steam bath until all the meat is dissolved (30–45 minutes). Add an equal volume of 95 per cent alcohol, cool and allow to stand for at least an hour. Filter by suction through a thin layer of asbestos in a Gooch crucible. Wash twice with a warm 4 per cent solution of potassium hydroxid in 50 per cent alcohol by volume and then twice with warm 50 per cent alcohol. Discard the washings. Retain as much of the precipitate in the beaker as possible until the last washing. Place the crucible with contents in the original beaker, add 40 cc. of water and 25 cc. of concentrated sulphuric acid. Stir during the addition of the acid and make sure that the acid comes in contact with all the precipitate. Allow to stand about 5 minutes, add 40 cc. of water and heat just to boiling, stirring constantly. Transfer the solution to a 250 cc. graduated flask, add 2 cc. of 20 per cent phosphotungstic acid solution, allow to cool to room temperature and make up to the mark with water. Filter through a starch-free filter paper, pipette 100 cc. of the filtrate into a 200 cc. graduated flask, neutralize with sodium hydroxid solution, make up to volume and determine the dextrose present in a 50 cc. portion of the filtrate, as directed under **VII, 25**, titrating the cuprous oxid precipitate as directed under **VII, 28**. The weight of the dextrose, multiplied by 0.9, gives the weight of the starch.

GLYCOGEN.

16 *Qualitative Test[5].—Tentative.*

Boil 50 grams of the macerated sample with 50 cc. of water for 15–30 minutes. Filter the broth through moistened filter paper or fine linen. To a portion of the filtrate in a test tube add a few drops of a mixture of 2 parts of iodin, 4 of potassium iodid and 100 of water. In the presence of a considerable amount of glycogen the latter produces a dark brown color, which is destroyed by heating and reappears on cooling. When starch is present, it may be precipitated by treating the water extract with two volumes of glacial acetic acid, filtering and applying the test for glycogen to the filtrate.

Quantitative Method[6].—Tentative.

17 PREPARATION OF SOLUTION.

Weigh out by difference about 25 grams of the finely ground and thoroughly mixed sample. Place in a 400 cc. beaker and mix with 50 cc. of potassium hydroxid solution (1.5 to 1), free from carbonate. Cover the beaker with a watch glass and digest on a water bath for 2 hours, with occasional stirring. At the end of the 2 hours, dilute to approximately 200 cc. with cold water.

Add to the solution an equal volume of 95 per cent alcohol by volume, cover with a watch glass and set aside for 10–12 hours. Decant the supernatant liquid through a folded 18.5 cm. filter, allowing the glycogen to remain in the beaker and wash by decantation with 66 per cent alcohol (2 volumes of 95 per cent alcohol to 1 of water) until the glycogen is white, or nearly so. Usually about 4 washings are required. Transfer the washed precipitate from the beaker to the filter and wash 2 or 3 times with 66 per cent alcohol. The solution filters slowly and the funnel should be covered with a watch glass to prevent excessive evaporation. The albuminous substance present retards the filtration if permitted to dry on the paper. If the washing by decantation is not made nearly complete, it will be difficult to obtain the glycogen free from the coloring matter.

After the washing is completed, close the bottom of the funnel by a piece of rubber tubing and a pinch-cock. Fill the funnel with warm water, cover with the watch glass and let stand 2–3 hours, or overnight. Open the pinch-cock and allow all of the solution to pass through the filter into a beaker. Close the funnel with the pinch-cock and fill with warm water as before. Allow this water to remain in the funnel for an hour and then filter as before. At first the glycogen solution appears quite turbid. This washing with warm water should be continued until the filtrate becomes perfectly clear. To the solution of glycogen in water, add double its volume of 95 per cent alcohol by volume and let stand overnight to complete the reprecipitation of the glycogen. Filter and wash as before with 66 per cent alcohol.

18 DETERMINATION.

If desired, the last filtration may be made through a tared Gooch crucible and the weight of glycogen determined after drying to constant weight. This gives results that are approximately correct. More satisfactory results are obtained by hydrolyzing the glycogen with dilute hydrochloric acid and determining the resultant dextrose. Dissolve the glycogen on the filter in warm water as directed above, collecting the filtrate and washings in a 300 cc. graduated flask and keeping the volume within 225 cc. Add 12.5 cc. of hydrochloric acid (sp. gr. 1.19) to the combined filtrate and washings, mix and place in a boiling water bath for 3 hours. Cool, neutralize with sodium hydroxid solution, cool again, make up to volume with water and determine dextrose in an aliquot of the solution as directed under **VII, 53**, determining the reduced copper as directed under **VII, 28**. Multiply the corresponding weight of dextrose by 0.9 to obtain its equivalent of glycogen and correct this result for dilution to obtain the per cent of glycogen in the sample.

SUGAR.—TENTATIVE.

19 REAGENT.

Phosphotungstic acid solution.—Dissolve 100 grams of phosphotungstic acid in water and dilute to 100 cc.

20 DETERMINATION.

Weigh 100 grams of the finely ground sample into a 600 cc. beaker, add 200 cc. of water, heat to boiling and boil gently for 5 minutes. Stir the contents of the beaker frequently during this and subsequent extractions to prevent bumping. (When several samples are extracted at the same time a mechanical stirring device is practically a necessity.) Remove the beaker from the flame, allow the insoluble matter to settle and decant the clear liquid on an asbestos mat in a 4-inch funnel. Filter with the aid of suction. Add 150 cc. of hot water to the residue in the beaker, boil gently for 5 minutes, let settle and decant the clear liquid as above. Repeat the operation and finally transfer the contents of the beaker to the funnel, wash with 150-200 cc. of hot water and press the meat residue as dry as possible. Transfer the contents of the filter flask to an evaporating dish and evaporate on a steam bath to a volume of about 25 cc., but not to dryness. Transfer the extract to a 100 cc. volumetric flask, taking care that the volume of liquid does not exceed 60 cc. Add 25-35 cc. of the phosphotungstic acid solution, shake vigorously, let stand a few minutes for gas bubbles to rise to the surface, make to volume, shake and either filter or centrifugalize. The use of a centrifuge is to be preferred since thereby a larger volume of liquid is obtained. Test a portion of the filtrate with dry phosphotungstic acid for complete precipitation. If an appreciable precipitate forms, take an aliquot of the filtrate, add 5-10 cc. of the phosphotungstic acid solution, make to volume, filter and test the filtrate for complete precipitation. The filtrate should also show not more than a slight reaction for creatinin by Jaffe's test[7].

Transfer 50 cc. of the clarified extract to a 100 cc. volumetric flask, add 5 cc. of concentrated hydrochloric acid and invert the solution as directed under **VII, 14.** Cool the solution, neutralize to litmus, cool, make to volume and filter. To the filtrate add sufficient dry powdered potassium chlorid to precipitate the excess of phosphotungstic acid, filter, test the filtrate for complete precipitation, and determine the reducing sugar, as directed under **VII, 25**, ascertaining the amount of reduced copper, as directed under **VII, 29**. Calculate the total sugar as dextrose.

If when the clarified meat extract is boiled with Fehling's solution an abnormal reduction is obtained, i. e., the solution turns yellow, brown, green or muddy in appearance instead of reddish-blue, the determination should be discarded, since incomplete precipitation of the nitrogenous compounds, due to the use of insufficient phosphotungstic acid, is indicated.

21 PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX.**

22 METALS.—TENTATIVE.

Proceed as directed under **XI.**

23 COLORING MATTERS.—TENTATIVE.

Proceed as directed under **X.**

SOLUBLE AND INSOLUBLE NITROGEN.—TENTATIVE.

24 PREPARATION OF SOLUTION.

Exhaust 7-25 grams of the sample (depending upon the water content) in the following manner: Weigh into a 150 cc. beaker, add 5-10 cc. of cold (15°C.) ammonia-free water and stir to a homogeneous paste. Then add 50 cc. of cold water, stir every 3 minutes for 15 minutes, let stand for 2-3 minutes and decant the liquid upon a quantitative filter, collecting the filtrate in a 500 cc. graduated flask. Drain the beaker, pressing out the liquid from the meat residue by the aid of a glass rod. Add to the residue in the beaker 50 cc. of cold water, stir for 5 minutes and, after standing 2-3 minutes, decant as before. If a considerable portion of the meat is carried over onto the filter, transfer it back to the beaker by means of a glass rod. Repeat the extractions, using the following additional amounts of cold water: 50, 50, 25, 25, 25 and 25 cc. After the last extraction transfer the entire insoluble portion to the filter and wash with three 10 cc. portions of water, allowing the material to drain thoroughly after each addition of water. Dilute to the mark and mix thoroughly.

25 DETERMINATION.

Determine the total nitrogen in a 50 cc. aliquot of the solution obtained under **24**, proceeding as directed under **I, 18, 21** or **23**. Subtract the percentage of soluble nitrogen from the percentage of total nitrogen, **6**, to obtain the percentage of insoluble nitrogen. To obtain the percentage of insoluble protein multiply the percentage of insoluble nitrogen by 6.25.

26 COAGULABLE NITROGEN.—TENTATIVE.

(For uncooked meat only.)

Measure 150 cc. of the extract, from **24**, into a 250 cc. beaker and evaporate to 40 cc. on a steam bath, with occasional stirring. Neutralize to phenolphthalein, then add 1 cc. of N/1 acetic acid and boil gently for 5 minutes. The coagulum should separate out at once, leaving a clear liquid. Filter on quantitative paper, wash the beaker thoroughly four times with hot water, taking special care to clean the sides. Finally wash the coagulum on the filter three times, dilute the combined filtrate and washings to a definite volume and reserve for the determination of proteoses, peptones and gelatin, **27**, and creatin, **29**. Transfer the coagulum with the paper to a Kjeldahl flask and remove, with concentrated sulphuric acid, any of the material adhering to the beaker, taking the usual 25 cc. of acid in 5 cc. portions for this purpose, heating the acid in the beaker on a hot plate and rubbing with a glass rod. Proceed as directed under **6**. Multiply the percentage of nitrogen obtained by 6.25 to obtain the percentage of coagulable proteins.

PROTEOSE, PEPTONE AND GELATIN NITROGEN.

27 *Modified Tannin-Salt Method*[8].—*Tentative.*

Transfer a 50 cc. aliquot of the filtrate, obtained in **26**, to a 100 cc. graduated flask, add 15 grams of sodium chlorid and 10 cc. of cold water, shake until the sodium chlorid has dissolved and cool to 12°C. Add 30 cc. of 24 per cent tannin solution, cooled to 12°C., fill to the mark with water previously cooled to 12°C., shake and allow the mixture to stand at a temperature of 12°C. for 12 hours or overnight. Filter at 12°C., transfer 50 cc. of the filtrate to a Kjeldahl flask and add a few drops of sulphuric acid. Place the flask in a steam bath, connect with a vacuum pump and evaporate to dryness. Determine nitrogen in the residue as directed under **I, 18**, using 30 cc. of sulphuric acid for the digestion. Conduct a blank determination, using the same amount of reagents, and correct the result accordingly. Multiply the corrected result by 2 and deduct the amount of nitrogen as found from the nitrogen determined in another 50 cc. aliquot of the filtrate from the coagulable nitrogen, **26**, without the tannin-salt treatment; the difference, multiplied by 6.25, gives the percentage of proteoses, peptones and gelatin.

28 MEAT BASES.—TENTATIVE.

Deduct from the percentage of total nitrogen, **6**, the sum of the percentages of nitrogen, obtained in the determination of insoluble nitrogen, **25**, coagulable nitrogen, **26**, and proteoses, peptones and gelatin, **27**, to obtain the percentage of nitrogen of the meat bases. Multiply the result by 3.12 to obtain the percentage of meat bases.

29 CREATIN.—OFFICIAL.

Evaporate an aliquot or the remaining portion of the filtrate and washings from the coagulable nitrogen, **26**, (a portion having been used in **27**), to 5–10 cc., transfer with a minimum amount of hot water to a 50 cc. measuring flask, keeping the volume below

30 cc., add 10 cc. of 2 N hydrochloric acid and mix. Hydrolyze in an autoclave at 117°–120°C. for 20 minutes, allow the flask to cool somewhat, remove and chill under running water. Partially neutralize the excess of acid by adding 7.5 cc. of 10 per cent sodium hydroxid solution, free from carbonates, dilute to the mark and mix. Make a preliminary reading on 20 cc. to ascertain the volume to use to obtain a reading of approximately 8 mm. and transfer to a 500 cc. graduated flask. Add 10 cc. of 10 per cent sodium hydroxid solution and 30 cc. of saturated picric acid solution (1.2 per cent). Mix and rotate for 30 seconds and let stand exactly 4.5 minutes. Dilute to the mark at once with water, shake thoroughly and read in a Duboscq colorimeter, comparing the color with N/2 potassium dichromate solution, set at 8 mm.

If the reading is too high or too low (above 9.5 or below 7 mm.), calculate the quantity necessary to obtain a reading of about 8 mm. The strength of the dichromate solution used must be checked against a standard creatin solution. To obtain the values, divide 81 by the reading and multiply by the volume factor to obtain mg. of creatinin; this value, multiplied by 1.16, gives creatin, which, divided by the weight of the sample and multiplied by 100, gives the per cent of creatin.

The use of Kober's shade and the painting of the plunger, suggested for this nephelometer, assists in getting a sharper end point, relieves the eye strain, and may be employed if desired.

Example.—Twenty grams of meat are extracted with water as directed under **24**, and the extract diluted to 500 cc.; 150 cc. of this latter solution (equivalent to 6 grams of meat) are treated as in **26**. The filtrate thus obtained is then evaporated and hydrolyzed as above and then diluted to 50 cc.; 25 cc. of this last solution are treated with sodium hydroxid solution and picric acid solution as directed above and diluted to 500 cc. This latter solution gives a Duboscq reading of 9 mm.

$$\frac{81}{9} \times \frac{50}{25} = 18 \text{ mg. creatinin;}$$

$$\frac{0.018 \times 1.16 \times 100}{6} = 0.35\% \text{ creatin.}$$

AMINO NITROGEN.

Van Slyke Method[9]*.—Tentative.*

30 REAGENTS.

(**a**) *Alkaline permanganate solution.*—Dissolve 50 grams of potassium permanganate and 25 grams of potassium hydroxid in sufficient water to make 1 liter.

(**b**) *Sodium nitrite solution.*—Dissolve 30 grams of sodium nitrite in sufficient water to make 100 cc.

(**c**) *Glacial acetic acid.*

31 APPARATUS.

Employ the apparatus shown in Figs. 8 and 9, the former illustrating the manner in which the entire apparatus is arranged and the latter showing the details of the deaminizing bulb and connections. The Hempel gas pipette is filled with the alkaline permanganate solution.

32 DETERMINATION.

Fill with water the burette (*F*), the capillary tube leading to the Hempel pipette and also the other capillary as far as *c*. Introduce into *A* sufficient glacial acetic acid to fill one-fifth of *D*, the tube (*A*) being etched with a mark to measure this amount.

Allow the acid to run into *D*, the cock *c* being turned so as to allow the air to escape from *D*. Pour the sodium nitrite solution into *A* until *D* is full of solution and enough excess is present to rise a little above the cock into *A*. *A* is also marked for measuring off this amount. Then close the gas exit from *D* at *c*, and, *a* being open, shake *D* for a few seconds until the liquid is forced down to the 20 cc. mark in *D*. Then close *a*, open *c* and shake the apparatus rapidly with the motor for 2 minutes, these operations being for the purpose of expelling all the air from *D*. Then turn *c* and *f* so that *D* and *F* are connected.

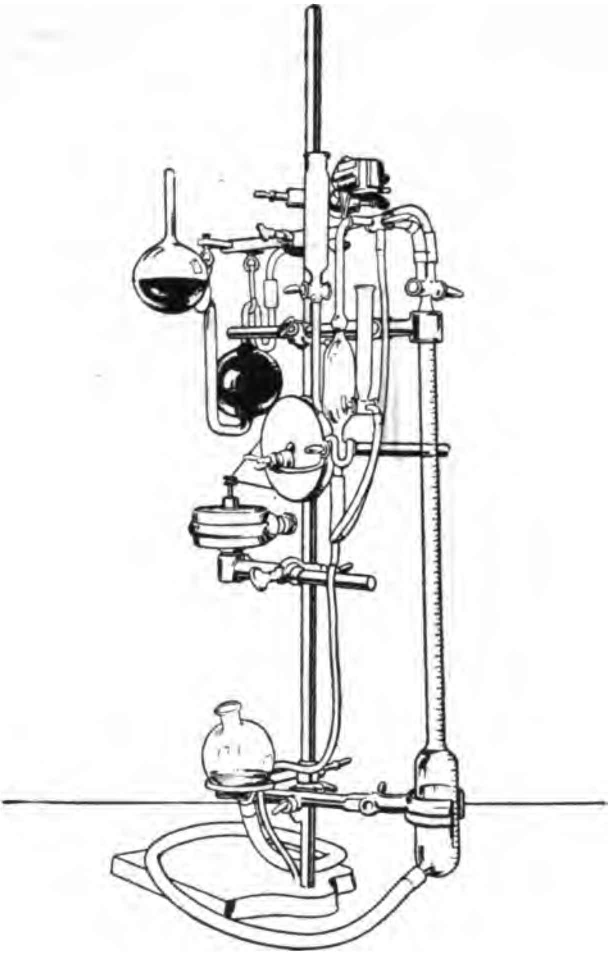

FIG. 8. VAN SLYKE APPARATUS FOR THE DETERMINATION OF AMINO NITROGEN. (*By courtesy of the Journal of Biological Chemistry.*)

Measure off in *B* 10 cc. or less, as the case may be, of the solution of the sample containing not more than 20 mg. of amino nitrogen (about 1–2 grams of the sample in the case of meat extracts) and allow it to run into *D*. Connect *D* with the motor as shown in Fig. 8 and shake for 5 minutes.

If the solution of the sample is viscous and threatens to foam over, rinse out *B*, and then through it introduce a little caprylic alcohol into *D*, or, if it is known beforehand that the sample will cause excessive foaming, introduce a little caprylic alcohol into *D* through *B*, rinsing *B* with alcohol and ether or drying with a roll of filter paper before adding the solution of the sample.

During the shaking there is an evolution of nitrogen mixed with nitric oxid, the gases being collected in *F*. Force all the gas in *D* into *F* by opening *a* and filling *D* with liquid from *A*. Connect *F* with the Hempel pipette and force the gas into the latter by means of the leveling bulb, allowing the cock *a* to remain open during this and the succeeding operation in order to permit displacement of the liquid in *D* by the nitric oxid formed in the interval. Connect the driving rod with the pipette by lifting the hook from the shoulder of *D* and placing the other hook, on the opposite side of the driving rod, over the horizontal lower tube of the pipette. Shake the pipette rather slowly for a minute, which, with any but almost completely exhausted permanganate solutions, completes the absorption of nitric oxid. Then return the gas

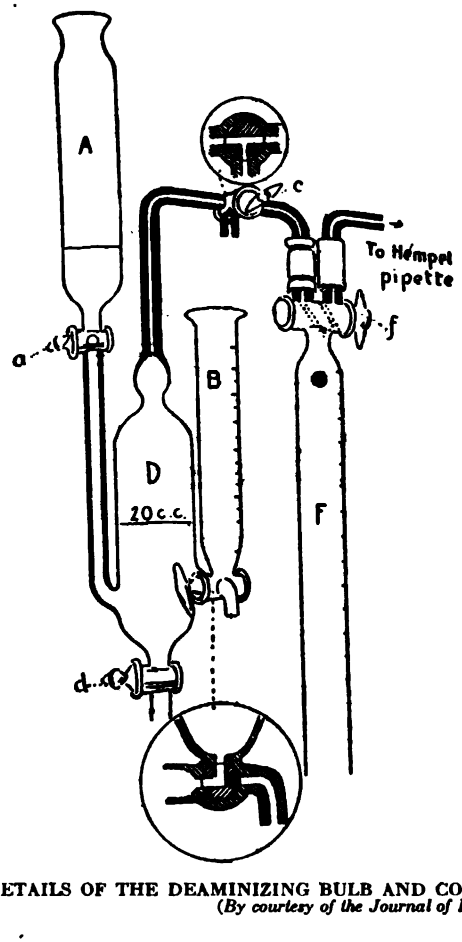

FIG. 9. DETAILS OF THE DEAMINIZING BULB AND CONNECTION.
(*By courtesy of the Journal of Biological Chemistry.*)

to the burette, adjust the level with the leveling bulb and note the volume of nitrogen, the temperature and barometric pressure, and calculate the volume of nitrogen under standard conditions of temperature and pressure. Obtain the corresponding weight of nitrogen, divide the latter by 2, and from the quotient calculate the apparent per cent of amino nitrogen in the sample. Correct the result for a blank test performed as above, using 10 cc. of water instead of the solution of the sample. The amount of gas obtained in the blank is usually 0.3–0.4 cc., and nitrite solutions giving a much larger correction should be rejected.

In the case of beef extracts and similar preparations, 5 minutes is sufficient time to allow for the completion of the reaction in *D*. In general the same time serves for the decomposition of alpha-amino acids, but with ammonia, methylamin and most amines other than alpha-amines 1–1.5 hours should be allowed. For determinations on such substances mix the solution of the sample with the reagents as described above, allow the mixture to stand in the apparatus till the end of the required time, and conclude the reaction by shaking the apparatus with the motor for 2–3 minutes. Continue the determination from this point as directed above.

33 *Sörenson Method*[10].—*Tentative.*

To 20 cc. of the filtrate from **26**, or 20 cc. of a solution containing an extract of the meat (in some cases a larger volume may be necessary) add 10 cc. of a freshly prepared phenolphthalein-formol mixture (50 cc. of commercial formol containing 1 cc. of a 0.5 per cent solution of phenolphthalein in 50 per cent alcohol, exactly neutralized with N/5 barium or sodium hydroxid). Titrate the mixture with N/5 barium hydroxid solution until a distinct red color appears, add a slight known excess of N/5 barium hydroxid and titrate back to neutrality with N/5 hydrochloric acid. Conduct a blank titration with the same reagents, using 20 cc. of water in place of the solution to be tested. From the amount of N/5 barium hydroxid required to neutralize the mixture, corrected for the amount used in the blank titration, calculate the amount of amino nitrogen present (including ammonia if this has not been removed). One cc. of N/5 barium hydroxid is equivalent to 2.8 mg. of amino nitrogen.

34 TOTAL SOLUBLE PHOSPHORUS.—TENTATIVE.

Evaporate to dryness 50 cc. of the water extract prepared under **24**, moisten the residue with 10 cc. of concentrated sulphuric acid, add a few drops of nitric acid and heat on a hot plate until all the organic matter is destroyed. Add 100 cc. of water, boil for a few minutes and proceed as directed under **I, 6**.

35 SEPARATION OF SOLUBLE INORGANIC AND ORGANIC PHOSPHORUS.—TENTATIVE.

To 500 cc. of the extract prepared as directed under **24**, add 50 cc. of magnesia mixture [**I, 4** (**c**)] and proceed as directed under **37**.

SOLUBLE PHOSPHORUS IN BLOOD, BRAIN AND GLANDULAR ORGANS[11].—TENTATIVE.

36 PREPARATION OF SOLUTION.

(**a**) *Cold water extract of flesh.*—Weigh out 10–12 grams of fresh muscle and divide equally between two small beakers. Moisten the sample with a few cc. of water, and break up the lumps with a glass rod. Add 50 cc. of water to each beaker and stir the contents for 15 minutes. Allow the insoluble residue to settle for 3–5 minutes, decant the liquid through filters into beakers and add 25 cc. of water to each residue. Stir 7–8 minutes and, after allowing to settle, decant onto the same filter. Continue this treatment, using 25 cc. of water each time, until the filtrates measure about 230 cc. each. Allow the filters to drain completely between extractions. Whenever the major portion of the residue has been mechanically transferred to the filter, return it to the beaker. After the last extraction, transfer the entire contents of each beaker onto the filter and, when drained, wash twice with small quantities of water. Combine the two extracts.

(**b**) *Hot water-ammonium sulphate extract of blood.*—Weigh out 30–35 grams of fresh whole blood as caught from the animal into a porcelain mortar. Grind and transfer to a 400 cc. beaker with hot water. Make up to about 150 cc. with boiling water. Place over a flame, gradually bring to boiling, with constant stirring, then add 20 cc. of 20 per cent ammonium sulphate solution and continue boiling, with constant stirring, for about 10 minutes. Decant onto an 18 cm. filter paper, receiving the filtrate in an 800 cc. beaker. Transfer the coagulum from the filter, along with that remaining in the beaker, to a mortar. Grind to a smooth paste and transfer to a beaker with boiling 3⅓ per cent ammonium sulphate solution. Make up to about 50 cc. with the latter, stir for 8 minutes and again filter. Return the coagulum to the mortar and grind again, transferring to the beaker as before with boiling 3⅓ per cent ammonium sulphate solution. Repeat this process of 8 minute extractions of the coagulum in 3⅓ per cent ammonium sulphate solution and filtration as directed above, without further grinding, until the filtrate measures about 450 cc. Wash out each beaker twice with 8–10 cc. of hot 3⅓ per cent ammonium sulphate solution, transferring the coagulum and extract to the filter. Wash the coagulum twice with boiling 3⅓ per cent ammonium sulphate solution from a wash bottle. Always allow the filter to drain well between additions of extract or wash solutions.

(**c**) *Hot water-ammonium sulphate extract of liver.*—Weigh by difference from a closed weighing bottle 15–20 grams of finely ground liver into a 400 cc. beaker. Add a few cc. of cold water and beat up with a stirring rod to separate the particles of tissue. Add enough boiling water to make the volume about 150 cc., place over a flame and bring to boiling. Add 10 cc. of 20 per cent ammonium sulphate solution and continue to boil for 10 minutes. Allow to settle for a moment and decant the boiling hot liquid onto an 18 cm. filter. Add 50 cc. of boiling water and stir for 8 minutes, without further heating, and decant onto the filter again. Repeat this addition of 50 cc. of hot water, stirring and decanting 8 times, returning the coagulum to the beaker as soon as any considerable amount collects upon the filter. With the eighth portion of water transfer the entire contents of the beaker onto the filter and wash twice with hot water. Always allow the filter to drain well between additions of extract or wash water.

(**d**) *Hot water-ammonium sulphate extract of brain.*—Weigh out about 10 grams of brain into a 250 cc. beaker. Add a few cc. of water and work up the brain and water with a glass rod. Make up to about 100 cc. with boiling water, place over a flame, and gradually bring to boiling, with constant stirring. While boiling vigorously (not before) add 20 cc. of 20 per cent ammonium sulphate solution, boil gently for about 10 minutes, allow to settle for a moment and decant slowly onto a linen filter containing acid-washed, glassmaker's sand, receiving the extract in an 800 cc. beaker. Add to the beaker containing the coagulum 50 cc. of 3⅓ per cent ammonium sulphate solution, stir 1 minute, keep boiling and decant the liquid onto the filter. Repeat this process of 1 minute extractions of the coagulum in 3⅓ per cent ammonium sulphate solution and filtration as directed above until the filtrate measures about 450 cc. Wash out the beaker twice with 8–10 cc. of hot 3⅓ per cent ammonium sulphate solution, completing the transfer of the coagulum and extract to the sand, and wash the coagulum twice with the wash solution. Always allow the filter to drain well between additions of extract or wash solution.

In making extracts of brain, give careful attention to the handling of the sample. The coagulum is very soft. It should be stirred only enough to keep it in motion. If handled roughly in returning from the sand filter to the beaker, it becomes too finely divided and retains a great deal of liquid. To prevent the extract or the coagulum from coming into contact with the linen before passing through the sand, pour the extract slowly into a slight depression in the center of the sand or, better, into a thin film of absorbent cotton, 1½ inches in diameter, laid over a depression in the sand. The coagulum remains on the cotton and its return to the beaker is facilitated thereby. If the cotton is not broken up by needless stirring, it can be taken out of the beaker with a glass rod and returned to the sand each time a partial extract is to be filtered. Care is necessary to prevent loss through bumping, on account of sand in the beakers, during the final extractions. Each partial extract should be boiling hot at the time filtration begins.

37 DETERMINATION.

To the extracts, prepared as directed under **36**, add 50 cc. of magnesia mixture [**I, 4** (**c**)] and stir thoroughly. Allow to stand 15 minutes, add 25 cc. of ammonium hydroxid (sp. gr. 0.90), cover and allow to stand 3 days. Filter and wash the precipitate with 2.5 per cent ammonium hydroxid. Dissolve the precipitate on the filter paper and that remaining in the beaker in nitric acid (1 to 1) and hot water, receiving the solution in a 400 cc. beaker. Neutralize with ammonium hydroxid, make slightly acid with nitric acid, add 5 grams of ammonium nitrate and determine phosphorus as directed under **I, 6**.

MEAT EXTRACTS AND SIMILAR PRODUCTS.

38 PREPARATION OF SAMPLE.—OFFICIAL.

Remove liquid and semi-liquid meat extracts and similar preparations from the container and mix thoroughly before sampling. A little heating expedites the mixing of pasty extracts. In many liquid preparations a sediment forms which should be carefully removed from the bottom of the container and included in the sample. If the sample is in the form of cubes, grind 10–12 of the cubes in a mortar.

39 MOISTURE.—OFFICIAL.

Proceed as directed under **VII, 2**, employing about 2 grams of powdered preparations, about 3 grams of pasty preparations, or 5–10 grams of liquid extracts, according to the solid content. Dry the powdered preparations directly without admixture. Dissolve the pasty preparations in water and dry with sufficient ignited sand, asbestos or pumice stone to absorb the solution. When glycerol is present, proceed as directed under **VII, 3**.

40 ASH.—OFFICIAL.

Proceed as directed under **VII, 4**. Add sufficient water to pasty preparations to effect solution and evaporate to dryness in order that the solids may be distributed evenly over the bottom of the dish.

41 TOTAL PHOSPHORUS.—OFFICIAL.

Proceed as directed under **5**.

42 CHLORIDS.—OFFICIAL.

Dissolve about 1 gram of the sample, prepared as directed under **38**, in 20 cc. of 5 per cent sodium carbonate solution and proceed as directed under **II, 20**.

43 FAT.—TENTATIVE.

Transfer the residue from the determination of moisture to a continuous extraction apparatus and proceed as directed under **VII, 10**.

44 TOTAL NITROGEN.—OFFICIAL.

Proceed as directed under **I, 18, 21** or **23**.

45 AMMONIA.—TENTATIVE.

Introduce 1 gram of pasty extracts or 2–3 grams of fluid extracts into tube *B* of the Folin apparatus and proceed as directed under **8**.

46 INSOLUBLE NITROGEN[12].—TENTATIVE.

Dissolve 5 grams of powdered preparations, 8–10 grams of pasty extracts, or 20–25 grams of fluid extracts, in cold water. Filter and wash with cold water. Transfer the filter paper and contents to a Kjeldahl flask and determine nitrogen as directed under **I, 18, 21** or **23**. However, if a large amount of insoluble matter is present, transfer the weighed sample to a graduated flask, make up to a definite volume, shake thoroughly, filter through a folded filter and determine nitrogen in an aliquot of the filtrate. Deduct the percentage of nitrogen in the total filtrate from the percentage of total nitrogen, **44**, to obtain the percentage of nitrogen in the insoluble protein. Multiply this percentage of nitrogen by 6.25 to obtain the percentage of insoluble protein.

47 COAGULABLE NITROGEN.—TENTATIVE.

Prepare a solution of the sample as directed under **46**. Employ as large an aliquot of the filtrate from the insoluble nitrogen, **46**, as practicable, and neutralize to phenolphthalein by the addition of acetic acid or sodium hydroxid, whichever may be necessary, add 1 cc. of N/1 acetic acid, boil for 2–3 minutes, cool to room temperature, dilute to 500 cc. and pass through a folded filter.

Determine nitrogen in 50 cc. of the filtrate as directed under **I, 18, 21** or **23**. Ten times the percentage of nitrogen so obtained subtracted from the percentage of soluble nitrogen (total nitrogen minus the percentage of nitrogen occurring as insoluble nitrogen) gives the percentage of nitrogen present as coagulable nitrogen. Multiply this figure by 6.25 to obtain the percentage of coagulable protein in the sample.

48 PROTEOSES AND GELATIN[13].—TENTATIVE.

Evaporate the filtrate from **47** to a small volume and saturate with zinc sulphate (about 85 grams to 50 cc., avoiding such an excess as would later cause bumping). Let stand several hours, filter and wash the precipitate with saturated zinc sulphate solution, place the filter and precipitate in a Kjeldahl flask and determine nitrogen as directed under **I, 18, 21** or **23**. Or, if the precipitate is voluminous, which rarely happens, make up to a definite volume with saturated zinc sulphate solution, filter and determine the nitrogen in an aliquot of the filtrate, as directed under **I, 18, 21** or **23**, and subtract the nitrogen thus obtained from the nitrogen in the filtrate from the coagulable nitrogen to obtain the nitrogen of the precipitated protein (proteoses and gelatin).

49 GELATIN.—TENTATIVE.

Prepare a 50 per cent solution of the sample, using hot water. Allow to cool and place in an ice box for 2 hours. If gelatin is present, the solution will set.

The ratio of total creatinin to total nitrogen in a normal meat extract (1 : 1.5) assists in determining the presence of gelatin or gelatin derivatives. The ratio is decreased when gelatin or gelatin derivatives are present in any considerable amount.

50 AMINO NITROGEN.—TENTATIVE.

Proceed as directed under **32** or **33**, using an aliquot of the filtrate from **47**.

51 ACID ALCOHOL-SOLUBLE NITROGEN[14].—TENTATIVE.

Transfer 10 cc. of an aqueous solution of the sample (10 grams of the sample dissolved in sufficient water to make 100 cc.) or, if the sample is insoluble in water, 1 gram of the sample and 10 cc. of water, to a 200 cc. glass-stoppered measuring cylinder, add 1.2 cc. of 12 per cent hydrochloric acid, mix and add absolute alcohol to the 200 cc. mark. Mix thoroughly and set aside for several hours. If necessary make up to volume, filter, transfer 100 cc. of the filtrate to a Kjeldahl flask, evaporate the alcohol on a water bath and determine nitrogen in the residue as directed under **I, 18, 21** or **23**.

52 CREATIN.—OFFICIAL.

Dissolve about 7 grams of the sample in cold (20°C.) ammonia-free water in a 150 cc. beaker, transfer the solution to a 250 cc. measuring flask, dilute to the mark and mix thoroughly. Transfer a 20 cc. aliquot of this solution to a 50 cc. measuring flask and proceed as directed under **29**. Subtract from the combined creatinin value the equivalent of the pre-formed creatinin, **53**, and multiply the difference by 1.16 to convert into creatin. Express the result as per cent of creatin.

53 CREATININ.—OFFICIAL.

For creatinin in beef extract measure about 5 cc. of the solution employed in **52** into a 500 cc. measuring flask, add 10 cc. of 10 per cent sodium hydroxid solution and 30 cc. of the saturated picric acid solution (1.2 per cent), mix and rotate for 30 seconds. Allow to stand exactly 4.5 minutes, then dilute to the mark at once with water. Shake thoroughly and read the depth of color after standing. If the reading is less than 7 or more than 9.5 mm., repeat, calculating the quantity of solution necessary to obtain a reading of about 8 mm. Express the result as per cent of creatinin, making the calculations as indicated under **29**.

54 NITRATES.—TENTATIVE.

Proceed as directed under **11** or **13**.

55 GLYCEROL[15].—TENTATIVE.

Weigh 2 grams of a solid or 5 grams of a liquid preparation in a small lead dish or Hofmeister Schälchen containing 20 grams of ignited sand. Transfer the dish and its contents to a mortar containing more ignited sand and several grams of anhydrous sodium sulphate and mix thoroughly. Transfer the mixture, including the dish, to a Soxhlet apparatus which has a piece of cotton placed in the side arm to prevent solid particles from being siphoned over. Extract the entire mass with redistilled anhydrous acetone for 10 hours. Distil the acetone from the extract, carefully removing the last trace by means of a vacuum pump. Take up the residue in water, add 5 cc. of 10 per cent silver nitrate solution, make up to a volume of 100 cc., shake, allow to stand overnight, filter and determine glycerol in an aliquot of the filtrate as directed under **XVIII, 6**, beginning at the point "Add * * * 30 cc. of the strong potassium dichromate solution". With solid meat and yeast extracts a blank of 0.5–1.0 per cent is obtained in most cases.

56 SUGAR.—TENTATIVE.

Heat 20 grams of the sample with about 200 cc. of water on a steam bath until all soluble substances have gone into solution, cool and proceed from this point as directed under **20**. Reducing sugar up to 0.5 per cent may be present as a natural constituent of meat extracts.

57 PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX**.

58 METALS.—TENTATIVE.

Proceed as directed under **XI**.

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XXI. DAIRY PRODUCTS.

MILK.

1 SOLIDS.—OFFICIAL.

Heat 3–5 grams of the milk at the temperature of boiling water until it ceases to lose weight, using a tared, flat-bottomed dish of not less than 5 cm. diameter. If desired, previously place 15–20 grams of pure, dry sand in the dish. Cool in a desiccator and weigh rapidly to avoid absorption of hygroscopic moisture.

2 ASH.—OFFICIAL.

Weigh about 20 grams of the milk in a tared dish, add 6 cc. of nitric acid, evaporate to dryness and ignite at a temperature just below redness until the ash is free from carbon.

3 TOTAL NITROGEN.—OFFICIAL.

Place about 5 grams of the milk in a Kjeldahl digestion flask and proceed, without evaporation, as directed under **I, 18, 21** or **23**. Multiply the percentage of nitrogen by 6.38 to obtain the equivalent percentage of nitrogen compounds.

CASEIN.

(This determination should be made while the milk is fresh, or nearly so. When it is not practicable to make this determination within 24 hours, add 1 part of formaldehyde to 2500 parts of milk and keep in a cool place.)

4 *Method I.—Official.*

Place 10 grams of the milk in a beaker with 90 cc. of water at 40°–42°C. and add at once 1.5 cc. of 10 per cent acetic acid. Stir and let stand 3–5 minutes. Then decant on a filter, wash by decantation 2 or 3 times with cold water and transfer the precipitate to the filter. Wash once or twice on the filter. The filtrate should be clear, or very nearly so. If the first portions of the filtrate are not clear, repeat the filtration, after which complete the washing of the precipitate. Determine nitrogen in the washed precipitate and filter paper as directed under **I, 18, 21** or **23**, and multiply by 6.38 to obtain the equivalent of casein.

In samples of milk which have been preserved, the acetic acid should be added in small portions, a few drops at a time, with stirring, and the addition continued until the liquid above the precipitate becomes clear, or very nearly so.

5 *Method II.—Official.*

To 10 grams of the milk add 50 cc. of water at 40°C., then 2 cc. of alum solution saturated at 40°C., or higher. Allow the precipitate to settle, transfer to a filter and wash with cold water. Determine nitrogen in the precipitate and filter paper as directed under **I, 18, 21** or **23**, and multiply by 6.38 to obtain the equivalent of casein.

ALBUMIN.

6 *Method I.—Official.*

Exactly neutralize the filtrate, obtained in **4**, with sodium hydroxid solution, add 0.3 cc. of 10 per cent acetic acid and heat on a steam bath until the albumin is completely precipitated. Collect the precipitate on a filter, wash with cold water, determine the nitrogen as directed under **I**, **18**, **21** or **23**, and multiply by 6.38 to obtain the equivalent of albumin.

7 *Method II.—Official.*

To the filtrate obtained from the casein determination, **5**, add 0.3 cc. of 10 per cent acetic acid, boil until the albumin is completely precipitated and proceed as directed under **6**.

LACTOSE.

Optical Method.—Official.

8 REAGENTS.

(**a**) *Acid mercuric nitrate solution.*—Dissolve mercury in double its weight of nitric acid (sp. gr. 1.42) and dilute with an equal volume of water.

(**b**) *Mercuric iodid solution.*—Dissolve 33.2 grams of potassium iodid and 13.5 grams of mercuric chlorid in 20 cc. of glacial acetic acid and 640 cc. of water.

9 DETERMINATION.

Determine the specific gravity of the milk by means of a delicate hydrometer or, if preferred, a pycnometer. The quantity of sample to be taken for the determination varies with the specific gravity and is to be measured at the same temperature at which the specific gravity is taken. The volume to be measured will be found in Table 12 (**10**), which is based upon twice the normal weight of lactose (32.9 grams per 100 cc.) for the Ventzke sugar scale.

Place the quantity of milk indicated in **10** in a flask graduated at 102.6 cc. Add 1 cc. of the acid mercuric nitrate solution or 30 cc. of the mercuric iodid solution (an excess of these reagents does no harm), fill to the mark, shake, filter through a dry filter and polarize. It is not necessary to heat before polarizing. If a 200 mm. tube is used, divide the polariscope reading by 2 (or, if a 400 mm. tube is used, by 4) to obtain the per cent of lactose in the sample.

TABLE 12.

10 *Volumes of milk corresponding to a lactose double normal weight*[1].

SPECIFIC GRAVITY OF MILK	VOLUME OF MILK FOR A LACTOSE DOUBLE NORMAL WEIGHT (VENTZKE SCALE)	SPECIFIC GRAVITY OF MILK	VOLUME OF MILK FOR A LACTOSE DOUBLE NORMAL WEIGHT (VENTZKE SCALE)
	cc.		*cc.*
1.024	64.25	1.030	63.90
1.025	64.20	1.031	63.80
1.026	64.15	1.032	63.75
1.027	64.05	1.033	63.70
1.028	64.00	1.034	63.65
1.029	63.95	1.035	63.55
		1.036	63.50

11 *Gravimetric Method.—Official.*

Dilute 25 grams of the milk with 400 cc. of water in a 500 cc. graduated flask, add 10 cc. of copper sulphate solution [**VII**, **19** (**a**)] and about 7.5 cc. of a potassium hydroxid solution of such strength that one volume is just sufficient to precipitate completely the copper as hydroxid from one volume of the copper sulphate solution.

Instead of potassium hydroxid solution of this strength, 8.8 cc. of N/2 sodium hydroxid solution may be used. After the addition of the alkali solution the mixture must still have an acid reaction and contain copper in solution. Fill the flask to the 500 cc. mark, mix, filter through a dry filter and determine lactose in an aliquot of the filtrate as directed under **VII, 45** or **47**.

FAT.

12 *Roese-Gottlieb Method*[2].—*Official.*

Weigh 10–11 grams of the milk into a Röhrig tube or some similar apparatus, add 1.25 cc. of concentrated ammonium hydroxid (2 cc. if the sample is sour) and mix thoroughly. Add 10 cc. of 95 per cent alcohol by volume and mix well. Then add 25 cc. of washed ether and shake vigorously for 30 seconds, then 25 cc. of petroleum ether (redistilled slowly at a temperature below 60°C.) and shake again for 30 seconds. Let stand 20 minutes, or until the upper liquid is practically clear. Draw off as much as possible of the ether-fat solution (usually 0.5–0.8 cc. will be left) into a weighed flask through a small, quick-acting filter. The flask should always be weighed with a similar one as a counterpoise. Re-extract the liquid remaining in the tube, this time with only 15 cc. of each ether, shake vigorously 30 seconds with each and allow to settle. Draw off the clear solution through the small filter into the same flask as before and wash the tip of spigot, the funnel and the filter with a few cc. of a mixture of the 2 ethers in equal parts free from suspended water. For absolutely exact results the re-extraction must be repeated. This third extraction yields usually not more than about 1 mg. of fat if the previous ether-fat solutions have been drawn off closely. Evaporate the ethers slowly on a steam bath, then dry the fat in a boiling water oven to constant weight.

Confirm the purity of the fat by dissolving in a little petroleum ether. Should a residue remain, remove the fat completely with petroleum ether, dry the residue, weigh and deduct the weight. Finally correct this weight by a blank determination on the reagents used.

Babcock Method.—Official.

13 APPARATUS.

(a) *Standard Babcock test bottles.*—The standard Babcock test bottles for milk and cream shall be as follows:

(1) *8 per cent, 18 gram, 6 inch milk test bottle.*—The total per cent graduation shall be 8. The total height of the bottle shall be 150–165 mm. (5⅞–6½ inches). The capacity of the bulb up to the junction with the neck shall be not less than 45 cc. The graduated portion of the neck shall have a length of not less than 63.5 mm. (2½ inches) and the neck shall be cylindrical for at least 9 mm. below the lowest and above the highest graduation marks. The graduations shall represent whole per cents, halves and tenths of a per cent.

(2) *50 per cent, 9 gram, 6 inch cream test bottle.*—The total per cent graduation shall be 50. The total height of the bottle shall be 150–165 mm. (5⅞–6½ inches). The capacity of the bulb up to the junction with the neck shall be not less than 45 cc. The graduated portion of the neck shall have a length of not less than 63.5 mm. (2½ inches) and the neck shall be cylindrical for at least 9 mm. below the lowest and above the highest graduation marks. The graduations shall represent five per cents, whole per cents and halves of a per cent.

(3) *50 per cent, 9 gram, 9 inch cream test bottle.*—Same as (2) except that the total height of the bottle shall be 210–225 mm. (8¼–8⅞ inches).

(b) *Centrifuge.*

(c) *Pipettes.*—Graduated to deliver 17.6 cc. of water at 20°C. in 5–8 seconds.

(d) *Graduates.*—Capacity 17.5 cc., or a Swedish acid bottle delivering that amount.

14 CALIBRATION OF APPARATUS.

(a) *Graduation.*—The unit of graduation for all Babcock glassware shall be the true cc. (0.998877 gram of water at 4°C.).

With bottles, the capacity of each per cent on the scale shall be 0.20 cc.

With pipettes and graduates, the delivery shall be the intent of the graduation; and the graduation shall be read with the bottom of the meniscus in line with the mark.

(b) *Testing.*—The method for testing Babcock bottles shall be calibration with mercury (13.5471 grams of clean, dry mercury at 20°C., to be equal to 5 per cent on the scale), the bottle being previously filled to zero with mercury.

The mercury and cork, alcohol and burette, and alcohol and brass plunger methods may be employed for the rapid testing of Babcock bottles, but the accuracy of all questionable bottles shall be determined by calibration with mercury.

The method for testing pipettes and graduates shall be calibration by measuring in a burette the quantity of water (at 20°C.) delivered.

(c) *Limit of error.*—For standard Babcock milk bottles the error at any point of the scale shall not exceed 0.1 per cent.

For standard Babcock cream bottles the error at any point of the scale shall not exceed 0.5 per cent.

For standard milk pipettes the error shall not exceed 0.05 cc.

For acid measures the error shall not exceed 0.2 cc.

15 DETERMINATION.

Pipette 17.6 cc. of the carefully mixed sample into a test bottle and add 17.5 cc. of commercial sulphuric acid (sp. gr. 1.82–1.83). Mix and, when the curd is dissolved, centrifulgalize for 4 minutes at the required speed for the machine used. Add boiling water, filling to the neck of the bottle, and whirl for 1 minute; again add boiling water so as to bring the fat within the scale on the neck of the bottle, and, after whirling for 1 minute more, read the length of the fat column, making the reading at 57°–60°C., at which temperature the fat is wholly liquid. The reading gives directly the per cent of fat in the milk.

Details of the manipulation of the Babcock test and its application in the analysis of dairy products other than milk are described by Farrington and Woll[3], and Van Slyke[4].

ADDED WATER.

(In conjunction with the copper, acetic or sour serum refraction method, the determination of the ash of the sour serum or of the acetic serum should be made in all cases where the indices of refraction fall below the minimum limit so as to eliminate all possibility of abnormal milk.)

16 ACETIC SERUM.—OFFICIAL.

(a) *Zeiss immersion refractometer reading.*—To 100 cc. of milk at a temperature of about 20°C. add 2 cc. of 25 per cent acetic acid (sp. gr. 1.035) in a beaker and heat the mixture, covered with a watch glass, in a water bath for 20 minutes at a temperature of 70°C. Place the beaker in ice water for 10 minutes and separate the curd from the serum by filtering through a 12.5 cm. folded filter. Transfer about 35 cc. of the serum to one of the beakers that accompanies the control-temperature bath used in connection with the Zeiss immersion refractometer, and take the refractometer reading at exactly 20°C., using a thermometer graduated to tenths of a degree. A reading below 39 indicates added water; between 39 and 40, the addition of water is suspected.

(**b**) *Ash.*—Transfer 25 cc. of the serum to a flat-bottomed platinum dish and evaporate to dryness on a water bath. Then heat over a low flame (to avoid spattering) until the contents are thoroughly charred, place the dish in an electric muffle, preferably with pyrometer attached, and ignite to a white ash at a temperature not greater than 500°C. (900°F.). Cool and weigh. Express the result as grams per 100 cc. Results below 0.715 gram per 100 cc. indicate added water. The acetic serum ash, multiplied by the factor 1.021, equals the sour serum ash (dilution of the acetic serum being 2 per cent).

17 SOUR SERUM.—TENTATIVE.

(**a**) *Zeiss immersion refractometer reading*[6].—Allow the milk to sour spontaneously, filter and determine the immersion refractometer reading of the clear serum at 20°C. A reading below 38.3 indicates added water.

(**b**) *Ash*[6].—Determine the ash in 25 cc. of the serum, obtained in (**a**), as directed in **16** (**b**). Results below 0.730 gram per 100 cc. indicate added water.

18 ZEISS REFRACTOMETER READING OF COPPER SERUM.—TENTATIVE.

To one volume of copper sulphate solution (72.5 grams of copper sulphate per liter, adjusted if necessary to read 36 at 20°C. on the scale of the Zeiss immersion refractometer, or, to a specific gravity of 1.0443 at $\frac{20°\text{C.}}{4°}$) add 4 volumes of milk. Shake well and filter. Determine the Zeiss refractometer reading of the clear serum at 20°C. A reading below 36 indicates added water.

GELATIN.

19 *Qualitative Test.—Tentative.*

To 10 cc. of the milk add an equal volume of acid mercuric nitrate solution (mercury dissolved in twice its weight of nitric acid (sp. gr. 1.42) and this solution diluted to 25 times its volume with water), shake the mixture, add 20 cc. of water, shake again, allow to stand 5 minutes and filter. If much gelatin is present, the filtrate will be opalescent and can not be obtained quite clear. To a portion of the filtrate contained in a test tube, add an equal volume of saturated aqueous picric acid solution. A yellow precipitate will be produced in the presence of any considerable amount of gelatin, while smaller amounts will be indicated by a cloudiness. In the absence of gelatin the filtrate will remain perfectly clear.

20 PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX**. To test for salicylic or benzoic acid acidify 100 cc. of the milk with 5 cc. of hydrochloric acid (1 to 3), shake until curdled, filter and treat the clear filtrate as directed under **IX**, **2**, **3** or **8**.

To test for formaldehyde proceed as directed under **IX**, **17**, **18**, **19**, **20**, **21**, **22**, **23** or **24**, applying the test directly to the milk.

21 COLORING MATTERS.—TENTATIVE.

Warm about 150 cc. of milk in a casserole over a flame and add about 5 cc. of acetic acid, then slowly continue the heating nearly to the boiling point while stirring. Gather the curd, when possible, into one mass with a stirring rod and pour off the whey. If the curd breaks up into small flecks, separate from the whey by straining through a seive or colander. Press the curd free from adhering liquid, transfer to a small flask and macerate for several hours, preferably overnight, in about 50 cc. of ether, the flask being tightly corked and shaken at intervals. Decant the ether extract into an evaporating dish, remove the ether by evaporation and test the fatty residue for annatto as directed under **X, 24**.

The curd of an uncolored milk is perfectly white after complete extraction with ether, as is also that of a milk colored with annatto. If the extracted fat-free curd is distinctly colored an orange or yellowish color, a coal tar dye is indicated. In many cases upon treating a lump of a fat-free curd in a test tube with a little strong hydrochloric acid the color changes to pink, indicating the presence of a dye similar to aniline yellow or butter yellow or perhaps one of the acid azo yellows or oranges. In such cases, separate and identify the coloring matter present in the curd as directed under **X**. If aniline yellow, butter yellow, or any other oil-soluble dye is present, the greater part will be found in the ether extract containing the fat. In such cases proceed as directed under **X, 3**.

In some cases the presence of coal tar dyes can be detected by treating about 10 cc. of the milk directly with an equal volume of hydrochloric acid (sp. gr. 1.20) in a porcelain casserole, giving the dish a slight rotary motion. In the presence of some dyes the separated curd acquires a pink coloration.

CREAM.

22 SOLIDS.—OFFICIAL.

Proceed as directed under **1**, employing 2–3 grams of the sample.

23 ASH.—OFFICIAL.

Proceed as directed under **2**.

24 TOTAL NITROGEN.—OFFICIAL.

Proceed as directed under **3**.

LACTOSE.

25 *Gravimetric Method.—Official.*

Proceed as directed under **11**.

FAT.

26 *Extraction Method.—Official.*

Weigh 4–5 grams of the homogeneous sample into a Röhrig tube or similar apparatus, dilute with water to about 10.5 cc. and proceed as directed under **12**.

27 *Babcock Method.—Official.*

Weigh 9 or 18 grams, depending upon the fat content of the sample, into a standard Babcock cream bottle and proceed as directed under **15**.

28 GELATIN.—TENTATIVE.

Proceed as directed under **19**.

29 COLORING MATTERS.—TENTATIVE.

Proceed as directed under **X**, particularly **3** and **24** for the detection of oil-soluble coal tar dyes and annatto.

30 PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX**.

31 CONDENSED MILK (UNSWEETENED).

Dilute 40 grams of the homogeneous sample with 60 grams of water and proceed as directed under **1** to **15**, inclusive, correcting the results for the dilution, other tests being applied as directed under **19**, **20** and **21**.

SWEETENED CONDENSED MILK.

32 PREPARATION OF SAMPLE.—OFFICIAL.

If the can is cold, place it in water at 30°–35°C. until warm. Open, scrape out all milk adhering to the interior of the can and mix by transferring the contents to a dish sufficiently large to permit stirring thoroughly and make the whole mass homogeneous. Weigh 100 grams into a 500 cc. flask and make up to the mark with water. If the milk will not dissolve completely, weigh out each portion for analysis separately.

33 TOTAL SOLIDS.—OFFICIAL.

Use 10 cc. of the solution, prepared as directed under **32**, and proceed as directed under **1**, drying on either sand or asbestos fiber.

34 ASH.—OFFICIAL.

Evaporate 10 cc. of the solution, prepared as directed under **32**, to dryness on a water bath and ignite the residue as directed under **VII, 4**.

35 PROTEIN.—OFFICIAL.

Determine nitrogen as directed under **I, 18, 21** or **23**, using 10 cc. of the solution prepared as directed under **32**, without evaporation and multiply by 6.38 to obtain the equivalent of protein.

36 LACTOSE.—OFFICIAL.

Dilute 100 cc. of the solution, prepared as directed under **32**, in a 250 cc. flask to about 200 cc., add 6 cc. of Fehling's copper sulphate solution [**VII, 19** (a)] and make up to the mark. Filter through a dry filter and determine lactose as directed under **VII, 45** or **47**.

37 FAT OR ETHER EXTRACT.—OFFICIAL.

Weigh 4–5 grams of the homogeneous sample into a Röhrig tube or some similar apparatus, dilute with water to about 10.5 cc. and proceed as directed under **12**.

SUCROSE.—TENTATIVE.

38 REAGENT[7].

To 220 grams of yellow mercuric oxid add 300–400 cc. of water and sufficient nitric acid to form a clear solution (about 140 cc. will be enough), being careful to use the least possible excess of acid. Dilute to 800–900 cc. and add sodium hydroxid solution slowly and with constant shaking until a slight permanent precipitate is obtained. Dilute to 1 liter and filter. The solution tends to become acid with age due to the deposition of basic mercuric salts. For this reason dilute alkali should be added occasionally until a slight permanent precipitate is formed and the solution filtered.

39

DETERMINATION.

Introduce 50 cc. of the solution prepared as directed under **32** into a 100 cc. graduated flask, add 25 cc. of water, mix, add 5 cc. of the mercuric nitrate reagent, and shake thoroughly. Without delay, and while shaking constantly, add sufficient N/2 sodium hydroxid solution to render the mixture neutral to litmus paper, being careful to avoid an alkaline reaction (usually 12–13 cc. will be required). Make up to 100 cc. with water, mix thoroughly and filter through a dry paper. Polarize the filtrate in a 200 mm. tube, then invert at room temperature as directed under **VII, 14**, and polarize the inverted solution. Correct both readings for the volume occupied by the protein, **35**, and the fat, **37**, one gram of protein occupying a space of 0.8 cc. and one gram of fat, 1.075 cc. Calculate the per cent of sucrose by the following formula, using the corrected direct and invert readings obtained above.

$$S = \frac{100\,(a-b)}{142.35-\frac{t}{2}} \times \frac{26}{W} \text{ in which}$$

S = per cent of sucrose in the sample,
a = corrected direct polarization,
b = corrected invert polarization,
t = temperature of solution polarized,
W = weight of sample taken (10 grams).

BUTTER AND ITS SUBSTITUTES.

40

PREPARATION OF SAMPLE.—OFFICIAL.

If large quantities of butter are to be sampled, use a butter trier or sampler. Melt completely the portions thus drawn, 100–500 grams, in a closed vessel at as low a temperature as possible. When melted, cool and, at the same time, shake the mass violently until it is homogeneous and solidified sufficiently to prevent the separation of the water and fat. Then pour a portion into the vessel from which it is to be weighed. The sample should completely or nearly fill the vessel and should be kept in a cool place until analyzed.

41

MOISTURE.—OFFICIAL.

Weigh 1.5–2.5 grams of the sample into a flat-bottomed dish, having a surface of at least 20 sq. cm., dry at the temperature of boiling water and weigh at hourly intervals until the weight becomes constant. The use of clean, dry sand or asbestos is admissible.

ETHER EXTRACT.

42

Indirect Method.—Official.

Dissolve the dry butter, obtained in the moisture determination in which no obsorbent was used, in absolute ether or petroleum ether, transfer to a weighed Gooch crucible with the aid of a wash bottle filled with the solvent and wash until free from fat. Dry the Gooch and contents at the temperature of boiling water until the weight is constant and determine the fat by difference.

43 *Direct Method.—Official.*

From the dry butter, obtained in the determination of moisture, either with or without the use of an absorbent, extract the fat with anhydrous, alcohol-free ether, receiving the solution in a weighed flask. Evaporate the ether, dry the extract at the temperature of boiling water and weigh at hourly intervals until the weight is constant.

44 CASEIN, ASH AND CHLORIN.—OFFICIAL.

Cover the crucible, containing the residue from the fat determination by the indirect method, **42**, and heat gently at first, then raise the temperature gradually to just below redness. The cover may then be removed and heating continued until the contents of the crucible are white. The loss in weight represents casein, and the residue in the crucible, mineral matter. Dissolve this mineral matter in water slightly acidified with nitric acid and determine chlorin, either gravimetrically as directed under **I, 16 (a)**, or volumetrically as directed under **II, 17**.

45 SALT.—OFFICIAL.

Weigh in a counterpoised beaker 5–10 grams of butter, using portions of about 1 gram each from different parts of the sample. Add about 20 cc. of hot water and, after the butter is melted, transfer the whole to a separatory funnel. Insert the stopper and shake for a few moments. Let stand until all the fat has collected on the top of the water, then draw off the latter into a flask, being careful to let none of the fat globules pass. Again add hot water. rinsing the beaker, and repeat the extraction 10–15 times, using 10–20 cc. of water each time. The washings will contain all but a mere trace of the sodium chlorid originally present in the butter. Determine the amount in the whole or an aliquot of the liquid by titration with standard silver nitrate, using potassium chromate as an indicator.

EXAMINATION OF FAT.

46 PREPARATION OF SAMPLE.—OFFICIAL.

Melt the butter and keep in a dry place at about 60°C. for 2–3 hours or until the water and curd have entirely separated. Filter the clear, supernatant fat through a dry filter paper in a hot water funnel or in an oven at about 60°C. If the filtered liquid fat is not perfectly clear, refilter.

47 *Physical and Chemical Methods.*

Proceed as directed under **XXII**.

48 *Microscopic Method.—Official.*

Place on a slide a small portion of the fresh, unmelted sample taken from the inside of the mass, add a drop of pure olive oil, apply a cover-glass with gentle pressure, and examine with magnification of 120–150 diameters for crystals of lard, etc. Examine the same specimen with polarized light and a selenite plate without the use of oil. Pure fresh butter will show neither crystals nor a parti-colored field with selenite. Renovated butter or other fats melted and cooled and mixed with butter will usually present crystals and variegated colors with the selenite plate.

For further microscopic study dissolve in a test tube 3–4 cc. of the fat in 15 cc. of ether. Close the tube with a loose plug of cotton wool and allow to stand 12–24 hours at 20°–25°C. When crystals form at the bottom of the tube, remove with a pipette, glass rod or tube, place on a slide, cover and examine under a microscope. The crystals formed by later deposits may be examined in a similar way. Compare with crystals obtained in the same way from samples of known purity.

49 PRESERVATIVES.—OFFICIAL.

Proceed as directed under **IX**.

50 COLORING MATTERS.—TENTATIVE.

Pour about 2 grams of the filtered fat, dissolved in ether, into each of two test tubes. Into one of the tubes pour 1–2 cc. of hydrochloric acid and into the other about the same volume of dilute potassium hydroxid solution. Shake the tubes well and allow to stand. In the presence of azo dyes the test tube to which the acid has been added will show a pink to wine-red coloration, while the potash solution in the other tube will show no color. If, on the other hand, annatto or other vegetable color has been used, the potash solution will be colored yellow, while no color will be apparent in the acid solution.

General test.—Proceed as directed under **X**, particularly **3** and **24**, for the detection of oil-soluble coloring matters and annatto.

RENOVATED BUTTER AND OLEOMARGARINE.

51 *Foam Test.—Tentative.*

Heat 2–3 grams of the sample, in either a spoon or dish, over a free flame. True butter will foam abundantly, whereas process butter will bump and sputter, like hot grease, without foaming. Oleomargarine behaves like process butter, but chemical tests will determine whether the sample is oleomargarine or butter.

52 *Melted Fat Test.—Tentative.*

Melt 50–100 grams of butter or renovated butter at 50°C. The curd from butter will settle, leaving a clear supernatant fat; in the case of renovated butter, the supernatant fat remains more or less turbid.

CHEESE.

53 SELECTION AND PREPARATION OF THE SAMPLE.—OFFICIAL.

When the cheese can be cut, take a narrow, wedge-shaped segment reaching from the outer edge to the center of the cheese. Cut this into strips and pass three times through a sausage machine. When the cheese can not be cut, take the sample with a cheese trier. If only one plug can be obtained, take it perpendicular to the surface of the cheese at a point one-third the distance from the edge to the center and extending either entirely or half way through it. When possible, draw three plugs, one from the center, one from a point near the outer edge, and one from a point half way between the other two. For inspection purposes reject the rind, but for investigations requiring the absolute amount of fat in the cheese include the rind in the sample. Either grind the plugs in a sausage machine or cut them very finely and mix carefully, preferably the former.

54 MOISTURE.—TENTATIVE.

Place 2–3 grams of very short fiber asbestos (the long fiber may be made suitable by rubbing it through a fine sieve) in a flat-bottomed platinum dish, 6–7 cm. in diameter, and press the asbestos down firmly. Place in the dish a glass rod, about 5 mm. in diameter and slightly longer than the diameter of the dish. Ignite, cool and weigh the dish and contents. Then weigh into the dish 4–5 grams of the sample, prepared as directed under **53**, and mix the cheese and asbestos intimately with the glass rod until the mass is homogeneous. Leave the mass in as loose a condition as possible to facilitate the drying. Dry the mixture in an oven at 100°C. and weigh at 1–1.5 hour intervals until the weight becomes constant (three weighings are usually sufficient).

55 ASH AND SALT.—OFFICIAL.

The dry residue from the moisture determination may be used for the determination of ash. If the cheese be rich in fat, the asbestos will be saturated with it. Ignite cautiously to avoid spattering and remove the lamp while the fat is burning. When the flame has died out, complete the ignition in a muffle at low redness. When desired, the salt may be determined in the ash, as directed under **44**.

56 NITROGEN.—OFFICIAL.

Determine nitrogen as directed under **I**, **18**, **21** or **23**, using about 2 grams of cheese, and multiply the percentage of nitrogen by 6.38 to obtain the per cent of nitrogen compounds.

57 ACIDITY.—OFFICIAL.

To 10 grams of finely divided cheese add water at a temperature of 40°C. until the volume equals 105 cc., shake vigorously and filter. Titrate 25 cc. portions of the filtrate, representing 2.5 grams of the sample, with standard sodium hydroxid, perferably N/10, using phenolphthalein as an indicator. Express the result in terms of lactic acid.

58 COLORING MATTERS.—TENTATIVE.

Proceed as directed under **X**.

FAT.

59 *Gravimetric Method.—Official.*

Cover the perforations in the bottom of an extraction tube with dry asbestos felt, and place on this a mixture containing equal parts of anyhydrous copper sulphate and pure, dry sand to a depth of about 5 cm., packing loosely. Cover the upper surface of this material with a layer of asbestos. Place on this 2–5 grams of the sample and extract with anhydrous ether for 5 hours in a continuous extraction apparatus. Remove the cheese to a mortar and grind it with pure sand to a fine powder, return the mixed cheese and sand to the extraction tube, wash the mortar with ether, add the washings to the tube and continue the extraction for at least 10 hours.

60 *Schmidt-Bondzynski Method, Modified.—Tentative.*

Rub up, by means of a glass rod, 1 gram of the homogeneous sample with 9 cc. of water, and 1 cc. of concentrated ammonium hydroxid in a narrow 100–125 cc. beaker. Digest the mixture at a low heat until the casein is well softened, neutralize with concentrated hydrochloric acid, using litmus as an indicator and add 10 cc. more of concentrated hydrochloric acid. Add a pinch of sand to prevent bumping and boil gently for 5 minutes, keeping the beaker covered with a watch glass. Cool the solution, transfer to a Röhrig tube or some similar apparatus, rinse the beaker with 25 cc. of washed ethyl ether and shake well. Add 25 cc. of redistilled petroleum ether (b. p. below 65°C.), let the solutions separate and proceed from this point as directed under **12**.

61 *Babcock Method.—Tentative.*

Weigh about 6 grams of the cheese in a tared dish. Add 10 cc. of boiling water and stir with a rod until the cheese softens and an even emulsion is formed, preferably adding a few drops of strong ammonium hydroxid, and keep the beaker in hot water until the emulsion is nearly completed and the mass is free from lumps. If the sample is from a whole milk cheese, employ a Babcock cream bottle. After cooling, transfer the contents of the beaker to the test bottle by adding to the beaker about one-half of the 17.6 cc. of sulphuric acid usually employed in this test, stirring with a rod, and pouring carefully into the bottle, using the remainder of the acid in two portions for washing out the beaker. Then proceed as directed under **15**. Multiply the fat reading by 18 and divide by the weight of the sample taken to obtain the per cent of fat.

EXAMINATION OF FAT.

62 PREPARATION OF SAMPLE.—TENTATIVE.

(**a**) *Alkaline extraction.*—Treat about 300 grams of the cheese, cut into fragments the size of a pea, with 700 cc. of 5 per cent potassium hydroxid solution at 20°C. in a large, wide-necked flask, shaking vigorously to dissolve the casein. In 5-10 minutes the casein will be dissolved and the fat will rise to the surface in lumps. Collect the lumps of fat into as large a mass as possible by shaking gently. Pour cold water into the flask until the fat is driven up into the neck and remove it by suitable means. Wash the fat thus obtained with just sufficient water to remove the residue of the alkali which it may contain. The fat is not perceptibly attacked by the alkali in this treatment, is practically all separated in a short time and is then easily prepared for chemical analysis by filtering and drying as directed under **46**.

(**b**) *Acid extraction.*—Pass the cheese through a grinding machine, transfer to a large flask and cover with warm water, using 1 cc. for every gram of cheese. Shake thoroughly and add sulphuric acid (sp. gr. 1.82–1.825) slowly and in small quantities, shaking after each addition of acid. The total amount of acid used should be the same as the amount of water employed. Remove the fat, which separates after standing a few minutes, by means of a separatory funnel, wash free from acid, filter and dry as directed under **46**.

63 *Physical and Chemical Methods.*

Proceed as directed under **XXII**.

ICE CREAM (PLAIN).

64 PREPARATION OF SAMPLE.—TENTATIVE.

Allow the sample to soften at room temperature. Owing to the fact that melted butter fat separates out and tends to rise to the surface, it is not advisable to soften the ice cream by heating on a water bath or over a flame. Mix thoroughly by stirring with a spoon or egg beater or by pouring back and forth between beakers.

FAT.

65 *Roese-Gottlieb Method.—Tentative.*

Weigh 4 grams of the thoroughly mixed sample into a small dry beaker, add 3 cc. of water, thoroughly mix with a glass rod and transfer to a Röhrig tube or similar apparatus, washing out the remaining portion with the aid of an additional 3 cc. of water. Add 2 cc. of concentrated ammonium hydroxid, mix thoroughly, and heat in a water bath at 60°C. From this point proceed as directed under **12**, beginning with "Add 10 cc. of 95 per cent alcohol by volume and mix well".

66 *Harding-Parkin Method.—Tentative.*

Weigh 5 grams of the sample prepared as directed under **64**, transfer to a Wener-Schmidt extraction tube, add 5 cc. of acetic acid (25 per cent by volume) and warm the contents of the tube to about 50°C. in a water bath. When the protein has dissolved, add 12 cc. of carbon tetrachlorid and shake the tube vigorously for 2 minutes, then add 20 cc. of 95 per cent alcohol by volume and shake thoroughly, add 30 cc. of petroleum ether, shaking vigorously for 2 minutes, and an additional 15 cc. of the same ether and continue shaking 1 minute longer. Close the tube and let stand until the liquids have separated. Insert the blowing-off device and blow out the ether layer cautiously through a filter into a weighing flask, care being taken that none of the acid-alcohol layer is blown over. Place 5 cc. of petroleum ether in a small evaporating dish and gently draw it into the tube by suction applied to the blowing-off device. After the ether has mixed with the layer in the tube, blow off and filter as before. Add 5 cc. of carbon tetrachlorid to the contents of the tube, shake thoroughly, then add 30 cc. of petroleum ether and again shake thoroughly. Allow to stand until the layers have separated, blow off the ether layer and wash once as in the first instance. Repeat the above operation, using 5 cc. of carbon tetrachlorid and 30 cc. of petroleum ether. Wash the filter paper with small portions of petroleum ether. Evaporate the ether slowly, heat the flask in an oven at a temperature of 100°C. and weigh.

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XXII. FATS AND OILS.

1 PREPARATION OF SAMPLE.—OFFICIAL.

Melt solid fats and filter by means of a hot water funnel or similar apparatus. Make the different determinations on samples of this melted, homogeneous mass. Filter oils that are not clear. Keep oils and fats in a cool place and protected from light and air, otherwise they will soon become rancid. Weigh out at one time as many portions as are needed for the various determinations, using a small beaker or weighing burette.

SPECIFIC GRAVITY.

2 *At* $\frac{20°C.}{4°}$.—*Official.*

Determine the specific gravity of the oil at $\frac{20° C.}{4°}$ by means of a pycnometer as directed under **XV, 3.**

If the specific gravity of the oil is determined at other than standard temperature, the approximate specific gravity at 20°C. may be calculated by means of the following formula:

$G = G' + 0.0007\ (T - 20°C.)$, in which

G = specific gravity at 20°C.,

G' = specific gravity at $\frac{T° C.}{4°}$,

T = temperature at which the specific gravity was determined,

0.0007 = mean correction[1] for 1°C.

At the temperature of boiling water.—Official.

3 STANDARDIZATION OF FLASKS.

(**a**) Fill a tared, 25–30 cc. specific gravity flask with freshly boiled hot water. Place in a briskly boiling water bath for 30 minutes, replacing any evaporation from the flask by the addition of boiling water. Then insert the stopper, previously heated to 100°C., remove the flask, cool and weigh.

(**b**) The following formula may be used for calculating the weight of water (W^T) which a given flask will hold at T° (weighed in air with brass weights at the temperature of the room) from the weight of water (W^t) (weighed in air with brass weights at the temperature of the room) contained therein at t°:

$W^T = W^t \frac{d^T}{d^t}\ [1 + 0.000026\ (T - t)]$, in which

d^T = the density of water at T°,

d^t = the density of water at t°.

4 DETERMINATION.

Fill the dry flask with the dry, hot, freshly filtered fat, which should be entirely free from air bubbles, and keep in a water bath for 30 minutes at the temperature of boiling water. Insert the stopper, previously heated to 100°C., cool and weigh. Divide the weight of contained fat by the weight of contained water previously found to obtain the specific gravity.

The weight of water at boiling temperature must be determined under the barometric conditions prevailing at the time the determination is made.

INDEX OF REFRACTION.

5 General Directions.—Official.

Place the instrument in such a position that diffuse daylight or any form of artificial light can readily be obtained for illumination. Circulate through the prisms a stream of water of constant temperature.

Determine the index of refraction with any standard instrument, reading oils at 20°C. and fats at 40°C.

The readings of the Zeiss butyro-refractometer can be reduced to standard temperature by the following formula[2]:

$$R = R' + 0.55\ (T' - T) \text{ in which}$$

R = the reading reduced to temperature T,
R' = the reading at T'C.
T' = the temperature at which reading R' is made,
T = the standard temperature,
0.55 = correction in scale divisions for 1°C.

With oils the factor 0.58 is substituted in the formula for 0.55, since they have a higher index of refraction.

The readings of instruments, which give the index of refraction directly, can be reduced to standard temperature by substituting the factor 0.000365 for 0.55 in the formula. As the temperature rises the refractive index falls.

The instrument used may be standardized with water at 20°C., the theoretical refractive index of water at that tempertture being 1.3330. Any correction found should be made on all readings.

The index of refraction varies with the specific gravity and in the same direction. If the results appear abnormal, compare the specific refractive power[3] with the normal. Calculate the specific refractive power from the formula $\frac{N-1}{D}$, in which N equals the refractive index and D the specific gravity. According to Proctor[4], the Lorenz formula $\frac{N^2-1}{(N^2+2)\,D}$ gives much more satisfactory results than $\frac{N-1}{D}$.

6 By Means of the Abbé Refractometer.—Official.

To charge the instrument, open the double prism by means of the screw head and place a few drops of the sample on the prism or, if preferred, open the prisms slightly by turning the screw head and pour a few drops of the sample into the funnel-shaped aperture between the prisms. Then close the prisms firmly by tightening the screw head. Allow the instrument to stand for a few minutes before the reading is made, so that the temperature of the sample and the instrument will be the same.

The method of measurement is based upon the observation of the position of the *border line of total reflection* in relation to the faces of a prism of flint glass. Bring this border line into the field of vision of the telescope by rotating the double prism by means of the alidade in the following manner: Hold the sector firmly, move the alidade backward or forward until the field of vision is divded into a light and a dark portion. The line dividing these portions is the "border line". This, as a rule, will not be a sharp line but a band of color. The colors are eliminated by rotating the screw head of the compensator until a sharp, colorless line is obtained. The border line should now be adjusted so that it falls on the point of intersection of the cross hairs. Read the refractive index of the substance directly on the scale of the sector. Check the correctness of the instrument, as directed under **5**, or by means of the quartz plate which accompanies it, using monobromnaphthalene, and make the necessary correction in the reading.

7 *By Means of the Zeiss-Butyro-Refractometer.—Official.*

Place 2 or 3 drops of the filtered fat on the surface of the lower prism. Close the prisms and adjust the mirror until it gives the sharpest reading. If the reading be indistinct after running water of a constant temperature through the instrument for some time, the fat is unevenly distributed on the surfaces of the prism. As the index of refraction is greatly affected by temperature, care must be used to keep the latter constant. The instrument should be carefully adjusted by means of the standard fluid which is supplied with it. Convert the degrees of the instrument into refractive indices from (8), Table 13.

TABLE 13.

8 *Butyro-refractometer readings and indices of refraction.*

READING	INDEX OF REFRACTION	READING	INDEX OF REFRACTION	READING	INDEX OF REFRACTION	READING	INDEX OF REFRACTION
40.0	1.4524	50.0	1.4593	60.0	1.4659	70.0	1.4723
40.5	1.4527	50.5	1.4596	60.5	1.4662	70.5	1.4726
41.0	1.4531	51.0	1.4600	61.0	1.4665	71.0	1.4729
41.5	1.4534	51.5	1.4603	61.5	1.4668	71.5	1.4732
42.0	1.4538	52.0	1.4607	62.0	1.4672	72.0	1.4735
42.5	1.4541	52.5	1.4610	62.5	1.4675	72.5	1.4738
43.0	1.4545	53.0	1.4613	63.0	1.4678	73.0	1.4741
43.5	1.4548	53.5	1.4616	63.5	1.4681	73.5	1.4744
44.0	1.4552	54.0	1.4619	64.0	1.4685	74.0	1.4747
44.5	1.4555	54.5	1.4623	64.5	1.4688	74.5	1.4750
45.0	1.4558	55.0	1.4626	65.0	1.4691	75.0	1.4753
45.5	1.4562	55.5	1.4629	65.5	1.4694	75.5	1,4756
46.0	1.4565	56.0	1.4633	66.0	1.4697	76.0	1.4759
46.5	1.4569	56.5	1.4636	66.5	1.4700	76.5	1.4762
47.0	1.4572	57.0	1.4639	67.0	1.4704	77.0	1.4765
47.5	1.4576	57.5	1.4642	67.5	1.4707	77.5	1.4768
48.0	1.4579	58.0	1.4646	68.0	1.4710	78.0	1.4771
48.5	1.4583	58.5	1.4649	68.5	1.4713	78.5	1.4774
49.0	1.4586	59.0	1.4652	69.0	1.4717	79.0	1.4777
49.5	1.4590	59.5	1.4656	69.5	1.4720	79.5	1.4780

MELTING POINT OF FATS AND FATTY ACIDS.

Wiley Method.—Official.

9 REAGENT.

Alcohol-water mixture.—Specific gravity same as that of the fat to be examined. Prepare by boiling, separately, water and 95 per cent alcohol by volume for 10 minutes to remove the gases which may be held in solution. While still hot pour the water into a test tube until it is almost half full. Nearly fill the test tube with the hot alcohol, poured down the side of the inclined tube to avoid too much mixing. If the alcohol be added after the water has cooled, the mixture will contain so many air bubbles as to be unfit for use.

10 DETERMINATION.

Prepare disks of fat as follows: Allow the melted and filtered fat to fall a distance of 15–20 cm. from a dropping tube upon a piece of ice or upon the surface of cold mercury. The disks thus formed should be 1–1.5 cm. in diameter and should weigh about 200 mg. When solid remove the disks and allow to stand 2–3 hours in order to obtain the normal melting point.

Place a test tube, 30 by 3.5 cm., containing the alcohol-water mixture, in a tall beaker, 35 by 10 cm., containing ice and water, until the mixture is cold. Then drop a disk of fat into the tube and it will at once sink to a point where the density of the alcohol-water mixture is exactly equivalent to its own. Lower an accurate thermometer, which can be read to 0.1°C., into the test tube until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol-water mixture around the disk, stir gently with the thermometer. Slowly heat the water in the beaker, constantly stirring it by means of an air blast or other suitable device.

When the temperature of the alcohol-water mixture rises to about 6°C. below the melting point of the fat, the disk of fat begins to shrivel and gradually rolls up into an irregular mass. Lower the thermometer until the fat particle is even with the center of the bulb. Rotate the thermometer bulb gently and regulate the temperature so that about 10 minutes for the increment of the last 2°C. are required. As soon as the fat mass becomes spherical, read the thermometer. Remove the tube from the bath and again cool. Place in the bath a second tube containing the alcohol-water mixture. The test tube is of sufficiently low temperature to cool the bath to the desired point, ice water having been used for cooling. After the first or preliminary determination, regulate the temperature of the bath so as to reach a maximum of about 1.5°C. above the melting point of the fat under examination.

Do not allow the edge of the disk to touch the sides of the tube. If it does, make a new determination. Run triplicate determinations. The second and third results should agree closely.

11 *Capillary Tube Method*[5].—*Official.*

Draw the melted fat or fatty acids into a thin-walled capillary tube. Use a column of fat 1–2 cm. long, according to the length of the thermometer bulb. Seal one end of the tube and cool on ice 12–15 hours. Attach the capillary tube to the bulb of an accurate thermometer, graduated to 0.2°C., immerse in a large test tube of water surrounded by a beaker of water and heat very slowly. An apparatus similar to that indicated in Fig. 10 may be used. The temperature at which the substance becomes transparent is taken as the melting point.

TITER TEST.

Alcoholic or Aqueous Sodium Hydroxid Method.—Official.

12 APPARATUS.

Standard Thermometer.—The thermometer must have a zero mark, 0.1° graduations between 10° and 60°C., and auxiliary reservoirs at the upper end and between the 0° and 10° marks. The cavity in the capillary tube between the 0° and the 10° marks must be at least 1 cm. below the 10° mark, which must be about 3–4 cm. above the bulb, the total length of the thermometer being about 38 cm. The bulb should be about 3 cm. long and 6 mm. in diameter. The stem of the thermometer should be 6 mm. in diameter and made of the best thermometer tubing, with scale etched on the stem, the graduation to be clear cut and distinct. The thermometer should have been annealed for 75 hours at 450°C., and the bulb should be of Jena normal 16$^{\text{III}}$ glass, moderately thin, so that the thermometer will be quick-acting.

13

DETERMINATION.

Saponify 75 grams of the sample in a metal dish with 60 cc. of 30 per cent sodium hydroxid solution (36° Baumé) and 75 cc. of 95 per cent alcohol by volume or 120 cc. of water. Evaporate to dryness over a very low flame or on an iron or asbestos plate, stirring constantly. Dissolve the dry soap in a liter of boiling water and, if alcohol has been used, boil for 40 minutes to remove it, adding sufficient water to replace that lost in boiling. Liberate the fatty acids by adding 100 cc. of 30 per cent sulphuric acid (25° Baumé) and boil until they form a clear, transparent layer. Wash the fatty acids with boiling water until free from sulphuric acid, collect in a small beaker and place on a steam bath until the water has settled and the fatty acids are clear, then decant into a dry beaker, filter while hot and dry 20 minutes at 100°C. When dried, cool the fatty acids to 15°–20°C. above the expected titer and transfer to the titer tube, 25 by 100 mm. (1 by 4 inches) and made of glass about 1 mm. in thickness. Place in a 16 ounce wide-mouthed bottle of clear glass, 70 by 150 mm. (2.8 by 6 inches), fitted with a perforated cork, so as to hold the tube rigidly when in position. Suspend the

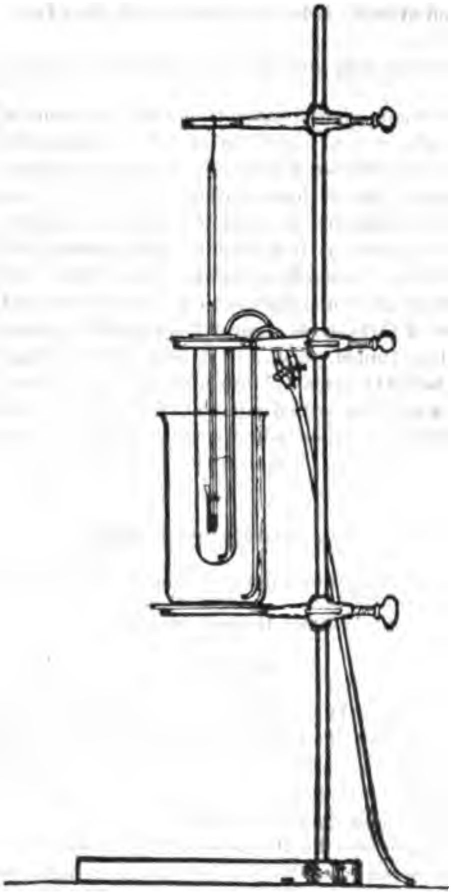

FIG. 10. APPARATUS FOR THE MELTING POINT DETERMINATION.

standard thermometer so that it can be used as a stirrer, and stir the mass slowly until the mercury remains stationary for 30 seconds. Then allow the thermometer to hang quietly, with the bulb in the center of the mass, and observe the rise of the mercury column. The highest point to which it rises is regarded as the titer of the fatty acids.

Test the fatty acids for complete saponification as follows:

Place 3 cc. in a test tube and add 15 cc. of 95 per cent alcohol by volume. Bring the mixture to a boil and add an equal volume of ammonium hydroxid (sp. gr. 0.96). A clear solution should result. The titer must be made at about 20°C. for all fats having a titer above 30°C., and at 10°C. below the titer for all other fats.

14 *Glycerol-Potassium Hydroxid Method.—Official.*

Heat 75 cc. of glycerol-potassium hydroxid solution (25 grams of potassium hydroxid in 100 cc. of high-test glycerol) to 150°C. in an 800 cc. beaker, then add 50 cc. of the oil or melted fat, previously filtered if necessary to remove foreign substances. Saponification often takes place almost immediately, but heating, with frequent stirring, should be continued for 15 minutes, avoiding a temperature much above 150°C. When the saponification is complete, as indicated by the perfectly homogeneous solution, pour the soap into an 800 cc. casserole containing about 500 cc. of nearly boiling water, add carefully 50 cc. of 30 per cent sulphuric acid and heat the solution, with frequent stirring, until the layer of fatty acids separates out perfectly clear. Transfer the fatty acids to a tall separatory funnel, wash 3 or 4 times with boiling water to remove all mineral acids, draw the fatty acids off into a small beaker, and allow to stand on a steam bath until the water has settled out and the acids are clear. Filter into a dry beaker and heat to 150°C. on a thin asbestos plate, stirring continually with the thermometer, transfer to a titer tube, fill it to within 2.5 cm. of the top and take the titer as directed under **13**.

IODIN ABSORPTION NUMBER.

(All reports of iodin absorption number should specifiy the method used.)

Hanus Method.—Official.

15 REAGENTS.

(**a**) *Hanus iodin solution.*—Dissolve 13.2 grams of pure iodin in 1 liter of glacial acetic acid (99.5 per cent) which shows no reduction with dichromate and sulphuric acid. Add enough bromin to double the halogen content as determined by titration (3 cc. of bromin are about the proper amount). The iodin may be dissolved by heating but the solution should be cold when the bromin is added.

A convenient way to prepare the Hanus solution is as follows: Measure 825 cc. of acetic acid which has shown no reduction by the dichromate test and dissolve in it 13.615 grams of iodin with the aid of heat. Cool and titrate 25 cc. of this solution against the N/10 sodium thiosulphate. Add 3 cc. of bromin to 200 cc. of acetic acid and titrate 5 cc. of the solution against the N/10 sodium thiosulphate. Calculate the quantity of bromin solution required exactly to double the halogen content of the remaining 800 cc. of iodin solution as follows:

$$A = \frac{B}{C} \text{ in which}$$

A = cc. of bromin solution required,
B = 800 × the thiosulphate equivalent of 1 cc. of iodin solution,
C = the thiosulphate equivalent of 1 cc. of bromin solution.

Example: 136.15 grams of iodin are dissolved in 8250 cc. of acetic acid. 30 cc. of bromin are dissolved in 2000 cc. of acetic acid. Titrating 50 cc. of the iodin solution against the standard thiosulphate shows that 1 cc. of the iodin solution equals 1.1 cc. of the thiosulphate (0.0165 gram of iodin). Titrating 5 cc. of the bromin solution shows that 1 cc. of the bromin solution equals 4.6 cc. of the thiosulphate. Then the quantity of bromin solution required to double the halogen content of the remaining 8200 cc. of iodin solution is equivalent to $\frac{8200 \times 1.1}{4.6}$ or 1961 cc. Upon mixing the two solutions in this proportion, a total volume of 10161 cc. is obtained, containing 135.3 grams of iodin. In order to reduce this solution to the proper strength (13.2 grams iodin per liter), $10.161 \times 13.2 = 134.1$; $135.3 - 134.1 = 1.2$ grams of iodin present in excess, or $\frac{1.2 \times 1000}{13.2} = 91$ cc. of acetic acid which must be added.

(b) *N/10 sodium thiosulphate solution.*—Prepare a solution containing 24.82 grams of sodium thiosulphate ($Na_2S_2O_3 5H_2O$) in water and dilute to 1 liter. Standardize this solution as follows: Place in a glass-stoppered flask 20 cc. of the N/10 potassium dichromate and 10 cc. of the 15 per cent potassium iodid solution. Add 5 cc. of strong hydrochloric acid. Dilute with 100 cc. of water and allow the N/10 sodium thiosulphate to flow slowly into the flask until the yellow color of the liquid has almost disappeared, add a few drops of the starch indicator and, with constant shaking, continue to add the N/10 sodium thiosulphate until the blue color just disappears.

(c) *Starch indicator.*—Prepare as directed under VI, 3 (a).

(d) *Potassium iodid solution.*—Dissolve 150 grams of potassium iodid in water and dilute to 1 liter.

(e) *N/10 potassium dichromate.*—Dissolve 4.903 grams of potassium dichromate in water and dilute to 1 liter. The strength of this solution should be checked against pure iron.

16 DETERMINATION.

Weigh about 0.500 gram of fat, or 0.250 gram of oil (0.100–0.200 gram in the case of drying oils which have a very high absorbent power), into a 500 cc. glass-stoppered flask or bottle. Dissolve the fat, or oil, in 10 cc. of chloroform. Add 25 cc. of the Hanus iodin solution and allow to stand for 30 minutes, shaking occasionally.

This time must be adhered to closely in order to obtain good results. The excess of iodin should be at least 60 per cent of the amount added. Add 10 cc. of the 15 per cent potassium iodid solution, shake thoroughly and then add 100 cc. of water, washing down any free iodin that may be found on the stopper. Titrate the iodin with the N/10 sodium thiosulphate, adding the latter gradually, with constant shaking, until the yellow color of the solution has almost disappeared. Add a few drops of the starch indicator and continue the titration until the blue color has entirely disappeared. Toward the end of the titration, stopper the bottle and shake violently, so that any iodin remaining in solution in the chloroform may be taken up by the potassium iodid solution. Conduct two blank determinations along with that on the sample. The number of cc. of the N/10 sodium thiosulphate required by the blank less the amount used in the determination gives the thiosulphate equivalent of the iodin absorbed by the fat or oil. Ascertain the iodin number by calculating the per cent by weight of iodin absorbed.

Wijs Method.—Tentative.

17 REAGENTS.

Wijs iodin solution.—Dissolve 13 grams of resublimed iodin in 1 liter of C. P. glacial acetic acid (99.0–99.5 per cent) and pass in washed and dried chlorin gas until the original thiosulphate titration of the solution is not quite doubled. Preserve in glass-stoppered amber bottles, sealed with paraffin until ready for use. Mark the date on which the solution is prepared on the bottle or bottles and do not use Wijs solution which is more than 30 days old.

There shall be no more than a slight excess of iodin and no excess of chlorin. When the solution is made from iodin and chlorin, this point can be ascertained by not quite doubling the titration.

The other reagents used are described under **15**.

18 DETERMINATION.

Weigh accurately 0.10–0.50 gram (depending on the iodin number) of the melted and filtered sample into a clean, dry, 16-oz. glass-stoppered bottle containing 15–20 cc. of carbon tetrachlorid or chloroform. Add 25 cc. of the iodin solution from a pipette, allowing to drain for a definite time. The excess of iodin should be from 50 to 60 per cent of the amount added, that is, from 100 to 150 per cent of the amount absorbed. Moisten the stopper with the 15 per cent potassium iodid solution to prevent loss of iodin or chlorin but guard against an amount sufficient to run down inside the bottle. Let the bottle stand in a dark place for 30 minutes at a uniform temperature. At the end of that time add 20 cc. of the 15 per cent potassium iodid solution and 100 cc. of water. Titrate the iodin with the N/10 sodium thiosulphate solution which is added gradually, with constant shaking, until the yellow color of the solution has almost disappeared. Add a few drops of the starch indicator and continue titration until the blue color has entirely disappeared. Toward the end of the reaction stopper the bottle and shake violently so that any iodin remaining in solution in the tetrachlorid or chloroform may be taken up by the potassium iodid solution. Conduct two determinations on blanks, which must be run in the same manner as the sample, except that no fat is used in the blanks. Slight variations in temperature quite appreciably affect the titer of the iodin solution, as acetic acid has a high coefficient of expansion. It is, therefore, essential that the blanks and determinations on the sample be made at the same time. The number of cc. of standard thiosulphate solution required by the blank, less the amount used in the determination, gives the thiosulphate equivalent of the iodin absorbed by the amount of sample used in the determination. Calculate to centigrams of iodin absorbed by 1 gram of sample (=per cent of iodin absorbed).

SAPONIFICATION NUMBER (KOETTSTORFER NUMBER).—OFFICIAL.

19 REAGENTS.

(**a**) *N/2 hydrochloric acid.*—Prepare as directed under **I, 16** (**a**).

(**b**) *Alcoholic potassium hydroxid solution.*—Dissolve 40 grams of the purest potassium hydroxid in 1 liter of 95 per cent redistilled alcohol by volume. The alcohol should be redistilled from potassium hydroxid over which it has been standing for some time, or with which it has been boiled for some time using a reflux condenser. The solution must be clear and the potassium hydroxid free from carbonates.

20 DETERMINATION.

Weigh accurately about 5 grams of the filtered sample into a 250–300 cc. Erlenmeyer flask. Pipette 50 cc. of the alcoholic potassium hydroxid solution into the flask, allowing the pipette to drain for a definite time. Connect the flask with an air condenser and boil until the fat is completely saponified (about 30 minutes). Cool and titrate with the N/2 hydrochloric acid, using phenolphthalein as an indicator. Calculate the

Koettstorfer number (mg. of potassium hydroxid required to saponify 1 gram of fat). Conduct a blank determination, using the same pipette and draining for the same length of time as above.

21 SOLUBLE ACIDS.—OFFICIAL.

Place the flask, used in **20**, and its contents on a water bath and evaporate the alcohol. Add such an amount of N/2 hydrochloric acid that its volume plus the amount used in titrating for the saponification number will be 1 cc. in excess of the amount required to neutralize the 50 cc. of the alcoholic potassium hydroxid solution added, and place on the steam bath until the separated fatty acids form a clear layer on the upper surface of the liquid. Fill to the neck with hot water and cool in ice water until the cake of fatty acids is thoroughly hardened. Pour the liquid contents of the flask through a filter into a liter flask. Fill the flask again with hot water, set on a steam bath until the fatty acids collect at the surface, cool by immersing in ice water, and again filter the liquid into the liter flask. Repeat this treatment with hot water three times, cooling and collecting the washings in the liter flask after each treatment. Titrate the combined washings with N/10 alkali, using phenolphthalein as an indicator. Subtract 5 (corresponding to the excess of 1 cc. of N/2 acid) from the number of cc. of N/10 alkali used and multiply by 0.0088 to obtain the weight of soluble acids as butyric acid. Calculate the percentage of soluble acids.

22 INSOLUBLE ACIDS (HEHNER NUMBER).—OFFICIAL.

Allow the flask, containing the cake of insoluble fatty acids from **21** and the paper through which the soluble fatty acids have been filtered, to drain and dry for 12 hours. Transfer the cake, together with as much of the fatty acids as can be removed from the filter paper, to a weighed, wide-mouthed beaker flask. Then place the funnel, containing the filter, in the neck of the flask and wash the paper thoroughly with hot absolute alcohol. Remove the funnel, evaporate the alcohol, dry for 2 hours at 100°C., cool in a desiccator and weigh. Again dry for 2 hours, cool and weigh. If there is any considerable decrease in weight, re-heat for 2 hours, cool and weigh again. Calculate the percentage of insoluble fatty acids.

SOLUBLE VOLATILE ACIDS (REICHERT-MEISSL NUMBER).

(As these determinations are entirely empirical, the directions given must be followed exactly. In reporting results the method used should always be stated.)

Reichert-Meissl Method.—Official.

23 REAGENTS.

(**a**) *Sodium hydroxid solution (1 to 1).*—The sodium hydroxid should be as free as possible from carbonates. Protect the solution from contact with carbon dioxid. Allow to settle and use only the clear liquid.

(**b**) *Potassium hydroxid solution.*—Dissolve 100 grams of the purest potassium hydroxid in 58 cc. of hot water. Cool in a stoppered vessel, decant the clear solution and protect from contact with carbon dioxid.

(**c**) *95 per cent alcohol by volume.*—Distilled over sodium hydroxid.

(**d**) *Dilute sulphuric acid.*—Dilute 200 cc. of the strongest acid to 1 liter with water.

(**e**) *Barium (or sodium) hydroxid solution.*—Standardize an approximately N/10 solution.

(**f**) *Indicator.*—Dissolve 1 gram of phenolphthalein in 100 cc. of 95 per cent alcohol.

(**g**) *Pumice stone.*—Heat small pieces to a white heat, plunge in water, and keep under water until used.

24 SAPONIFICATION.

Weigh 5.75 cc., about 5 grams, of the filtered sample, into a saponification flask and proceed by one of the following three methods:

(1) *Under pressure with alcohol.*—Place 10 cc. of the 95 per cent alcohol in the flask containing the fat (the flask must be made of strong, well-annealed glass, capable of resisting the tension of alcoholic vapor at 100°C.) and add 2 cc. of the sodium hydroxid solution. Insert a soft cork stopper into the neck of the flask, tie it down and place the stoppered flask on a water or steam bath for at least an hour, rotating the flask gently from time to time. Cool before opening.

(2) *Under pressure without the use of alcohol.*—Place 2 cc. of the potassium hydroxid solution in the flask containing the fat (the flask being round-bottomed and made of well-annealed glass to resist pressure), cork and heat as directed under (1). This method avoids the formation of esters and the removal of the alcohol after saponification.

(3) *With a reflux condenser and the use of alcohol.*—Place 10 cc. of the 95 per cent alcohol in the flask containing the fat, add 2 cc of the sodium hydroxid solution and heat on a steam bath until the saponification is complete, using a reflux condenser.

After the saponification, if alcohol was used, remove it by evaporation on a steam bath. Eliminate the last traces of alcohol by waving the flask briskly, mouth down, or better, by blowing out with a current of carbon dioxid-free air.

25 DISTILLATION AND TITRATION.

Dissolve the soap, obtained as directed under **24**, by adding 135 cc. of recently boiled water (132 cc. if potassium hydroxid was used in the saponification) and warm on the water bath, with occasional shaking, until the solution is clear. Cool to 60°–70°C., add 5 cc. of the dilute sulphuric acid (8 cc. if potassium hydroxid was used in the saponification), stopper loosely and heat on a water bath until the fatty acids form a clear, transparent layer. Cool to room temperature, add a few pieces of the pumice stone and connect with a glass condenser by means of a bulb tube. Heat slowly with a free flame until ebullition begins and distil, regulating the flame so as to collect 110 cc. of distillate in as nearly 30 minutes as possible. Mix this distillate, filter through a dry filter, and titrate 100 cc. with the standard barium or sodium hydroxid solution, using phenolphthalein as an indicator. The red color should remain unchanged for 2–3 minutes.

Multiply the number of cc. of N/10 alkali used by 1.1, divide by the weight of fat taken and multiply by 5 to obtain the Reichert-Meissl number. Correct the result by the figure obtained in a blank determination.

Leffman and Beam Method.—Official.

26 REAGENTS.

Glycerol-soda solution.—Add 20 cc. of the sodium hydroxid solution, prepared as directed under **23** (**a**), to 180 cc. of pure, concentrated glycerol.

The other reagents used are described under **23**.

27 DETERMINATION.

Add 20 cc. of the glycerol-soda solution to about 5 grams of the fat in a flask, weighed as directed under **24**, and heat over a free flame or on an asbestos plate until complete saponification takes place, as shown by the mixture becoming perfectly clear. If foaming occurs, shake the flask gently.

Add 135 cc. of recently boiled water, drop by drop at first, to prevent foaming, and 5 cc. of the dilute sulphuric acid, distil without previous melting of the fatty acids, using an apparatus similar to that illustrated in **28**, Fig. 11, regulating the flame so as to collect 110 cc. of distillate in as nearly 30 minutes as possible. Filter, titrate the volatile acids and calculate the Reichert-Meissl number, as directed under **25**. Conduct a blank determination and correct the result accordingly.

28 INSOLUBLE VOLATILE ACIDS[6] (POLENSKE NUMBER).—OFFICIAL.

Proceed as directed under **27** up to the point at which 110 cc. of distillate have been collected, except that only 20 minutes are allowed for the distillation, employing an apparatus of the *exact* dimensions illustrated in Fig. 11. Substitute a 25 cc. cylinder for the receiving flask to collect any drops that may fall after the flame has been removed. Immerse the flask containing the distillate almost completely in water at 15°C. for 15 minutes, filter the 110 cc. of distillate and determine the approximate Reichert-Meissl number, if desired, as directed under **27**, avoiding too violent shaking of the distillate and consequent emulsification of the insoluble acids previous to filtration. Remove the remainder of the soluble acids from the insoluble acids upon the filter paper by washing with three successive portions of 15 cc. of water, previously passed through the condenser, the 25 cc. cylinder and the 110 cc. receiving flask. Then dissolve the insoluble acids by passing three successive 15 cc. portions of neutral 90 per cent alcohol by volume through the filter, each portion previously passed through the condenser, the 25 cc. cylinder and the 110 cc. receiving flask. Titrate the combined alcoholic washings with N/10 sodium hydroxid, using phenolphthalein as an indicator.

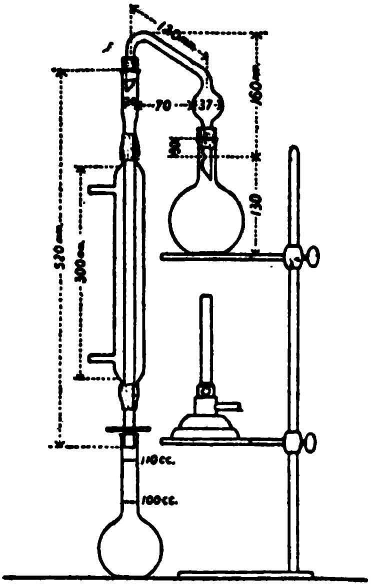

FIG. 11. APPARATUS[7] FOR THE DETERMINATION OF THE POLENSKE NUMBER.

Run a blank in the same manner and subtract the quantity of the standard alkali required to neutralize the 45 cc. of alcohol, used in washing the apparatus and filter paper of the blank, from that required in each Polenske determination. Report the Polenske number as the number of cc. of N/10 alkali required to neutralize the insoluble volatile acids from the 110 cc. of distillate obtained as above *upon a 5 gram sample*. Since the entire distillate is filtered it is not necessary to multiply the burette reading by 1.1, as in **25**, but a calculation must be made, as directed under **25**, to reduce the actual number of cc. found in the titration to the number which would have been required had exactly 5 grams of fat been used.

29 LIQUID AND SOLID FATTY ACIDS[8].—TENTATIVE.

Weigh 5 grams of the oil or fat into an Erlenmeyer flask, saponify, precipitate with lead acetate solution and treat the precipitated lead soap with ether, as directed under **37**. Filter the ether solution of the soluble lead soap into a Muter tube or separatory funnel and decompose the soap by shaking with 40 cc. of hydrochloric acid (1 to 5). The soap is completely decomposed when the ether becomes clear and colorless.

Draw off the lead chlorid from the ether solution and wash the ether free from acid. Using an atmosphere of carbon dioxid, in order to prevent the oxidation of the oleic acid, evaporate an aliquot of this solution until free from ether and weigh to determine the per cent of liquid acids. Determine the iodin number as directed under **16** or **18**, using 0.2–0.3 gram of this residue.

As it is very difficult to dry the unsaturated acids without very serious oxidation, it is just as satisfactory to determine the weight of insoluble acids by the following method:

Wash the insoluble soap left on the filter into a flask, decompose with hydrochloric acid and heat until the fatty acids are melted. Fill the flask with hot water, cool, pour off the water and again wash the solidified fatty acids. Dissolve in hot 95 per cent alcohol by volume, transfer to a weighed dish, remove the alcohol by evaporation, dry, weigh and calculate the per cent of solid fatty acids.

30 FREE FATTY ACIDS.—OFFICIAL.

Weigh 20 grams of fat, or oil, into a flask, add 50 cc. of 95 per cent alcohol by volume which has been neutralized with dilute sodium hydroxid solution, using phenolphthalein as an indicator, and heat to boiling. Shake the flask thoroughly in order to dissolve the free fatty acids as completely as possible. Titrate with N/10 alkali, shaking thoroughly until the pink color persists after vigorous shaking.

Express the results either as percentage of oleic acid, as acid degree (cc. of N/1 alkali required to neutralize the free acids in 100 grams of oil or fat), or as acid value (mg. of potassium hydroxid required to saturate the free acids in 1 gram of fat or oil).

One cc. of N/10 alkali is equivalent to 0.0282 gram of oleic acid.

31 ACETYL VALUE[9].—OFFICIAL.

Boil the oil or fat with an equal volume of acetic anhydrid for 2 hours, pour the mixture into a large beaker containing 500 cc. of water and boil for 30 minutes. To prevent bumping, pass a slow current of carbon dioxid into the liquid through a finely drawn out tube reaching nearly to the bottom. Allow the mixture to separate into two layers, siphon off the water, and boil the oily layer with fresh water until it is no longer acid to litmus paper. Separate the acetylated fat from the water and dry and filter in a drying oven.

Weigh 2–4 grams of the acetylated fats into a flask and saponify with alcoholic potash as directed under **20**. If the distillation process is to be adopted, it is not necessary to work with a standardized alcoholic potassium hydroxid solution, but in the filtration method, which is much shorter, the alcoholic potassium hydroxid solution must be measured exactly. In either case evaporate the alcohol after saponification and dissolve the soap in water. Then either distil or filter as follows:

(1) *Distillation.*—Acidify with sulphuric acid (1 to 10) and distil as directed under **25**. Since several hundred cc. must be distilled, either run a current of steam through or add portions of water from time to time (500–700 cc. of distillate will be sufficient). Filter the distillate to remove any insoluble acids carried over by the steam and titrate with N/10 potassium hydroxid, using phenolphthalein as an indicator. Multiply the number of cc. of alkali employed by 5.61 and divide by the weight of substance used to obtain the acetyl value.

(2) *Filtration.*—Add to the soap solution a quantity of standard sulphuric acid exactly corresponding to the amount of alcoholic potassium hydroxid solution added, warm gently, filter off the free fatty acids which collect on top, wash with boiling water until the washings are no longer acid and titrate the filtrate with N/10 potassium hydroxid, using phenolphthalein as an indicator. Calculate the acetyl value as directed under (1).

CHOLESTEROL AND PHYTOSTEROL IN MIXTURES OF ANIMAL AND VEGETABLE FATS.

32 *Alcohol Extraction Method*[10].—*Tentative.*

Introduce 200–300 grams of the melted fat into a flat-bottomed liter flask. Close the neck of the flask with a 3-holed stopper and insert through these holes: (1) A reflux condenser, (2) a right-angled glass tube, one arm of which reaches to a point 6 mm. above the surface of the melted fat, the other being closed a short distance from the flask by means of a short piece of rubber tubing and a pinch-cock, (3) a glass tube bent so that one arm reaches down to the bottom of the flask and the other serves as a delivery tube for a 700 cc. round-bottomed flask containing 500 cc. of 95 per cent alcohol by volume.

Place the flasks, containing the melted fat and the alcohol, on a steam bath and heat so that the alcohol vapor passes through the melted fat in the liter flask and is condensed in the reflux condenser, finally collecting in a layer over the melted fat. After all the alcohol has passed in this manner into the flask containing the fat, disconnect the flask from which the alcohol has been distilled and attach a tube to the short piece of rubber tubing attached to the right-angled glass tube [see (2) above] and siphon the alcohol layer back into the alcohol distillation flask. Reconnect as at first and again distil the alcohol as in the first operation. When all the alcohol has been distilled, siphon it again into the distillation flask and extract in the same manner a third time.

Discard the fat and retain the alcohol which now contains practically all of the cholesterol and phytosterol originally present in the fat. Concentrate the alcoholic solution to about 250 cc. and add 20 cc. of potassium hydroxid solution (1 to 1) to the boiling liquid. Boil for 10 minutes to insure complete saponification of the fat, cool to room temperature and pour into a large separatory funnel containing 500 cc. of warm ether. Shake to insure thorough mixing and add 500 cc. of water. Rotate the funnel gently to avoid the formation of extremely stubborn emulsions, but mix the water thoroughly with the alcohol-ether-soap solution. A clear, sharp separation takes place at once. Draw off the soap solution and wash the ether layer with 300 cc. of water, avoiding shaking. Repeat the washing of the ether solution with small quantities of water until all the soap is removed. Transfer the ether layer to a flask and distil the ether until the volume of liquid remaining in the flask measures about 25 cc. Transfer this residue to a tall 50 cc. beaker and continue the evaporation until all the ether is driven off and the residue is perfectly dry. If desired, a tared beaker may be used and the weight of the unsaponifiable matter determined at this point.

Add 3–5 cc. of acetic anhydrid to the residue in the beaker, cover the beaker with a watch glass and heat to boiling over a free flame. After boiling for a few seconds, remove the beaker from the flame, cool and add 35 cc. of 60 per cent alcohol by volume. Mix the contents of the beaker thoroughly, filter off the alcoholic solution and wash the precipitate with 60 per cent alcohol. Dissolve the precipitate on the filter with a stream of hot 80 per cent alcohol by volume and wash the insoluble portion well with 80 per cent alcohol. Acetates of cholesterol and phytosterol are dissolved while the greater portion of the impurities present (including paraffin and paraffin oil if present) remain behind on the filter. Cool the combined filtrate and washings to a temperature of 10°–12°C. and allow to stand at that temperature for 2–3 hours. During this time the acetates of cholesterol and phytosterol crystallize from the solution. Collect the crystals upon a filter, wash with cold 80 per cent alcohol and then dissolve them in a minimum amount of hot absolute alcohol. Collect the alcoholic solution of the acetates in a small, glass evaporating dish, add 2 or 3 drops of water to the solution and heat if not perfectly clear. Allow the alcohol to evaporate spontaneously, the contents of the dish being stirred occasionally to mix the deposit of crystals which form upon the edges with the main body of the liquid. As soon as a good deposit of crystals has formed, collect them upon a hardened filter, wash twice with cold 90 per cent alcohol and dry by suction, drying finally at 100°C. for 30 minutes, and determine the melting point in the apparatus shown in **11**, Fig. 10, using sulphuric acid in the outer beaker and glycerin in the inner tube.

The melting point of the first crop of crystals usually gives definite information as to the presence or absence of phytosterol but the conclusion indicated should be confirmed by recrystallizing the crystals from absolute alcohol and again determining the melting point. If the crystals are pure cholesteryl acetate, the melting point of the second crop should agree closely with that of the first. If phytosteryl acetate is present, however, a higher melting point will be noted, as phytosteryl acetate is less soluble in alcohol than cholesteryl acetate. The melting point of cholesteryl acetate is 114°C., that of phytosteryl acetate 125°–137°C.

33 *Digitonin Method*[11].—*Tentative.*

Shake vigorously 50 grams of the oil, or fat, for 15 minutes in a separatory funnel with 20 cc. of a 1 per cent solution of digitonin in 95 per cent alcohol by volume. Allow the mixture to stand for a time until the emulsion separates. The lower or fat layer should be quite clear while the alcohol layer contains a bulky, flocculent precipitate. Draw off as much as possible of the fat, avoiding any loss of the precipitate. Add 100 cc. of ether to the alcohol layer and filter the mixture. Wash the precipitate with ether until free from fat, after drying in the air, transfer it to a tall 50 cc. beaker, add 2–3 cc. of acetic anhydrid and cover the beaker with a watch glass. Then boil slowly over a low flame for 30 minutes. After cooling, add 30–35 cc. of 60 per cent alcohol by volume and mix the contents of the beaker thoroughly. Filter the alcohol solution and wash the precipitate with 60 per cent alcohol, then dissolve it on the filter with a stream of hot 80 per cent alcohol by volume from a wash bottle and set aside the filtrate in a cool place (10°C. or below). After the acetates have crystallized out of this solution filter them off, recrystallize from absolute alcohol, dry and determine the melting point of each crop of crystals, as directed under **32**.

34 UNSAPONIFIABLE RESIDUE[12].—OFFICIAL.

Saponify 5 grams of the oil, or fat, with alcoholic potassium hydroxid solution and remove the alcohol by evaporation. Wash into a separatory funnel with 70–100 cc. of water and extract with 50–60 cc. of ether. If the two liquids do not separate. add a few cc. of alcohol. Separate the water solution and wash the ether with water containing a few drops of sodium hydroxid solution. Again extract the soap solution and washings with ether and evaporate the combined extracts to dryness. In most cases it is advisable to add a little alcoholic potassium hydroxid solution to the residue and heat in order to saponify any traces of fats left unsaponified and extract again with ether. Transfer to a weighed dish and dry as quickly as possible in a water oven.

Many of the hydrocarbon oils are volatile at 100°C., so that the drying should not be carried any further than necessary. With resin oil, paraffin wax and the denser mineral oils there is little danger of loss at 100°C.

On account of the solubility of soap in ether and petroleum ether it is well to wash the residue with warm water containing a little phenolphthalein. If the reaction is alkaline, soap is present and the residue must be further purified.

RESIN OIL.

35 *Qualitative Test.—Tentative.*

Polarize the pure oil, or a definite dilution, with petroleum ether, in a 200 mm. tube. Resin oil has a polarization in a 200 mm. tube of from + 30° to + 40° on the sugar scale (Schmidt and Haensch) while most oils[13] read between + 1° and − 1°.

COTTONSEED OIL.

36 *Halpen Test[14].—Official.*

Mix carbon disulphid, containing about 1 per cent of sulphur in solution, with an equal volume of amyl alcohol. Mix equal volumes of this reagent and the oil under examination, and heat in a bath of boiling, saturated brine for 1–2 hours. In the presence of as little as 1 per cent of cottonseed oil, a characteristic red or orange-red color is produced.

Lard and lard oil from animals fed on cottonseed meal will give a faint reaction; their fatty acids also give this reaction.

The depth of color is proportional, to a certain extent, to the amount of oil present, and by making comparative tests with cottonseed oil some idea as to the amount present can be obtained. Different oils react with different intensities, and oils which have been heated to 200°–210°C.[15] react with greatly diminished intensity. Heating for 10 minutes at 250°C. renders cottonseed oil incapable of giving the reaction[16].

37 PEANUT OIL[17].—OFFICIAL.

Weigh 20 grams of the oil into an Erlenmeyer flask. Saponify with alcoholic potash solution, neutralize exactly with dilute acetic acid, using phenolphthalein as an indicator, and wash into an 800–1000 cc. flask containing a boiling mixture of 100 cc. of water and 120 cc. of 20 per cent lead acetate solution. Boil for a minute and then cool the precipitated soap by immersing the flask in water, occasionally giving it a whirling motion to cause the soap to stick to the sides of the flask. After the flask has cooled, decant the water and excess of lead acetate solution and wash the lead soap with cold water and 90 per cent alcohol by volume. Add 200 cc. of ether, cork and allow to stand for some time until the soap is disintegrated, heat on a water bath, using a reflux condenser, and boil for about 5 minutes[18]. In the case of oils, most of the soap will be dissolved, while in lards, which contain much stearin, part of the soap will be left undissolved. Cool the ether solution of soap to 15°–17°C. and allow to stand until all the insoluble soaps have separated out (about 12 hours).

Filter upon a Büchner funnel and thoroughly wash the insoluble lead soaps with ether. Wash the ether-insoluble lead soaps into a separatory funnel by means of a jet of ether, alternating at the end of the operation, if a little of the soap sticks to the paper, with hydrochloric acid (1 to 3). Add sufficient hydrochloric acid (1 to 3) so that the total volume of the latter amounts to about 200 cc. and enough ether to make its total volume 150–200 cc. and shake vigorously for several minutes. Allow the layers to separate, run off the acid layer, and wash the ether once with 100 cc. of dilute hydrochloric acid and then with several portions of water until the water washings are no longer acid to methyl orange. If a few undecomposed lumps of lead soap remain (indicated by solid particles remaining after the third washing with water), break these up by running off almost all the water layer and then add a little concentrated hydrochloric acid, shake and continue the washing with water as before. Distil the ether from the solution of insoluble fatty acids and dry the latter in the flask by adding a little absolute alcohol and evaporating on a steam bath. Dissolve the dry fatty acids by warming with 100 cc. of 90 per cent alcohol by volume. Cool slowly to 15°C., shaking to aid crystallization. Allow to stand at 15°C. for 30 minutes. In the presence of peanut oil, crystals of arachidic acid will separate from the solution. Filter, wash the precipitate twice with 10 cc. of 90 per cent alcohol by volume, and then with 70 per cent alcohol by volume, care being taken to maintain the arachidic acid and the wash solutions at a definite temperature in order to apply the solubility corrections given below. Dissolve the arachidic acid upon the filter with boiling absolute alcohol, evaporate to dryness in a weighed dish, dry and weigh. Add to the weight 0.0025 gram for each 10 cc. of 90 per cent alcohol used in the crystallization and washing, if conducted at 15°C.; if conducted at 20°C., add 0.0045 gram for each 10 cc. The melting point of arachidic acid thus obtained is 71°–72°C. Twenty times the weight of arachidic acid will give the approximate amount of peanut oil present. Arachidic acid has a characteristic appearance and may be identified under the microscope. As little as 5–10 per cent of peanut oil can be detected by this method.

SESAME OIL.

38

Baudouin Test.—Official.

Dissolve 0.1 gram of finely powdered sugar in 10 cc. of hydrochloric acid (sp. gr. 1.20), add 20 cc. of the oil to be tested, shake thoroughly for a minute and allow to stand. The aqueous solution separates almost at once and, in the presence of even a very small admixture of sesame oil, is colored crimson. Some olive oils give a slight pink coloration with this reagent. Comparative tests with known samples containing sesame oil will differentiate them.

39

Villavecchia Test[19]*.—Official.*

Add 2 grams of furfural to 100 cc. of 95 per cent alcohol by volume and mix thoroughly 0.1 cc. of this solution, 10 cc. of hydrochloric acid (sp. gr. 1.20), and 10 cc. of the oil by shaking them together in a test tube. A crimson color is developed as in the Baudouin test, 38, where sugar is used.

Villavecchia explained this reaction on the basis that furfural is formed by the action of levulose and hydrochloric acid and therefore substituted furfural for sucrose. As furfural gives a violet tint with hydrochloric acid it is necessary to use the very dilute solution specified in the method.

DETECTION OF FOREIGN FATS CONTAINING TRISTEARIN IN LARD.

40

Method I[20]*.—Tentative.*

Weigh 5 grams of the melted fat into a glass-stoppered 25 cc. cylinder about 150–175 mm. in height. Add warm ether up to the 25 cc. mark, stopper securely and shake until the fat is completely dissolved. Allow the cylinder to stand for about 18 hours at a temperature of 16°–20°C., during which time some of the solid glycerides will crystallize out. Decant the clear solution carefully from the crystals, wash with three 5 cc. portions of cold ether, being careful to avoid breaking up the deposit during the first two washings. Agitate the crystals with the third portion of ether and transfer to a small filter. Wash on the paper with successive small amounts of cold ether until 15–20 cc. have been used, then remove the last traces of ether by means of slight suction on the stem of the funnel. Break up any large lumps and allow the deposit to dry.

When thoroughly dry pulverize the glycerides and take their melting point in a closed 1 mm. tube, using an apparatus similar to that indicated in **11**, Fig. 10. Heat the water in the beaker rapidly to about 55°C. and maintain that temperature until the thermometer carrying the melting-point tube registers 50°–55°C., then heat again and carry the temperature of the outer bath somewhat rapidly to 67°C., when the lamp is removed. The melting point of the crystals is regarded as that point when the fused substance becomes perfectly clear and transparent. A dark background placed about 4 inches from the apparatus will prove of advantage. When the melting point of the glycerides obtained by this method is below 63.4°C. the presence of beef fat should be suspected, while a melting point of 63°C., or below, can be regarded as positive evidence that the sample is not pure lard. It is advisable to carry out this method with a control sample of pure lard in connection with each batch of samples analyzed.

41 *Method II.—Tentative.*

Weigh 5 grams of the filtered fat into a glass-stoppered cylinder graduated to 25 cc., and add warm acetone until the 25 cc. mark is reached. Shake the cylinder until the contents are thoroughly mixed, and allow to stand in a suitable place in which a temperature of 30°C. is maintained. After 18 hours remove the cylinder and carefully decant the supernatant acetone solution from the crystallized glycerides, which are usually found in a firm mass at the bottom of the cylinder. Add warm acetone in three portions of 5 cc. each from a small wash bottle, care being taken not to break up the deposit while washing, and decant the first two portions. The third portion is, however, actively agitated in the cylinder, and by a quick movement transferred with the crystals to a small filter paper. Wash the crystals with five successive small portions of the warm acetone, using the wash bottle, and remove the excess acetone from them by suction. Transfer the paper with its contents to a suitable place, where it should be spread out and any large lumps of the glycerides broken up by gentle pressure. When dry, thoroughly comminute the mass and determine the melting point of the crystals in a closed 1 mm. tube as directed under **40**. A melting point below 63°C. is regarded as evidence of adulteration and a melting point below 63.4°C. is regarded as suspicious.

After the melting point of the crystallized glycerides has been determined, transfer them to a 50 cc. beaker, add 25 cc. of approximately N/2 alcoholic potassium hydroxid and heat on a steam bath until saponification is complete. Pour the solution into a separatory funnel containing 200 cc. of water, acidify, add 75 cc. of ether and shake. Draw off the acid layer and wash at least three times with water. Transfer the ether solution to a clean, dry 50 cc. beaker, drive off the ether on the steam bath and finally dry the acids at 100°C. After the acids have stood for at least 2 hours, after drying, determine the melting point in the same manner in which the melting point of the crystals was determined. If the melting point of the glycerides, plus twice the difference between the melting point of the glycerides and melting point of the fatty acids, is less than 73°C. the lard is regarded as adulterated.

FISH OIL AND MARINE ANIMAL OILS IN THE PRESENCE OF VEGETABLE OILS AND IN THE ABSENCE OF METALLIC SALTS.

42 *Qualitative Test.—Tentative.*

Dissolve in a test tube about 6 grams of the oil in 12 cc. of a mixture of equal parts of chloroform and glacial acetic acid. Add bromin, drop by drop, until a slight excess is indicated by the color, keeping the solution at about 20°C. Allow to stand 15 minutes or more and then place the test tube in boiling water. If vegetable oils only are present, the solution will become perfectly clear, while fish oils will remain cloudy or contain a precipitate due to the presence of insoluble bromids.

43 COLORING MATTERS.—TENTATIVE.

Proceed as directed under X. 3.

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XXIII. SPICES AND OTHER CONDIMENTS.

SPICES.

1

PREPARATION OF SAMPLE.—OFFICIAL.

Grind the sample to pass through a sieve having circular openings 1 mm. in diameter and mix thoroughly. Owing to the lack of uniformity of most spices and the peculiar tendency to stratify, extreme care is necessary in weighing out a portion for analysis. Stir the material with a spoon having a capacity of approximately 2 grams and dip a spoonful from the interior in order that only a very small amount needs to be added to or taken from the portion on the scale pan. In the determination of starch in spices by the diastase method reduce a portion of the sample to an impalpable powder by grinding in a mortar.

2

MOISTURE.—TENTATIVE.

Dry 2 grams to constant weight at 110°C. From the resulting loss in weight subtract the amount of volatile ether extract as determined under **9**.

3

ASH.—OFFICIAL.

Determine as directed under **VII, 4**.

4

SOLUBLE AND INSOLUBLE ASH.—OFFICIAL.

Proceed as directed under **VIII, 14**, employing the ash obtained as directed under **3**.

5

ASH INSOLUBLE IN ACID.—OFFICIAL.

Boil the water-insoluble residue, obtained as directed under **4**, or the total ash obtained as directed in **3**, with 25 cc. of 10 per cent hydrochloric acid (sp. gr. 1.050) for 5 minutes, collect the insoluble matter on a Gooch crucible or an ashless filter, wash with hot water, ignite and weigh.

6

CALCIUM OXID IN ASH.—OFFICIAL.

Ignite 2–4 grams of the sample as directed under **3**, digest with hot 10 per cent hydrochloric acid, evaporate to dryness, moisten the dry residue with dilute hydrochloric acid and again evaporate to dryness to render the silica insoluble. Moisten the residue with 5–10 cc. of hydrochloric acid, add about 50 cc. of water, allow to stand on a water bath for a few minutes, filter and wash the insoluble residue with hot water. Determine calcium oxid in the combined filtrate and washings as directed under **XXVII, 23**.

7

NITROGEN.—OFFICIAL.

Determine as directed under **I, 18, 21** or **23**, except in the case of black and white peppers, for which use only the Kjeldahl-Gunning-Arnold method[1] [**I, 23**], employing 1 gram of the sample.

8

NITROGEN IN NON-VOLATILE ETHER EXTRACT.—OFFICIAL.

(For black and white peppers.)

Extract 10 grams of the pepper for 20 hours in a continuous extraction apparatus with absolute ether, collecting the extract in a weighed 250 cc. flask. Evaporate the ether, dry first at 100°C. and finally to constant weight at 110°C. Determine the nitrogen in the weighed extract, as directed under **I, 23**, digesting in the same flask used for the extraction. Calculate the parts of nitrogen per 100 parts of non-volatile ether extract. If desired, crude piperin may be calculated from the nitrogen by multiplying by 20.36.

9 VOLATILE AND NON-VOLATILE ETHER EXTRACT[2].—OFFICIAL.

Extract 2 grams of the ground material for 20 hours in a continuous extraction apparatus with anhydrous ether [**VII, 9**]. Transfer the ethereal solution to a tared capsule and allow to evaporate at room temperature. Let stand for 18 hours over sulphuric acid and weigh the total ether extract. Heat the extract gradually and then to constant weight at 110°C. The loss is volatile ether extract; the residue, non-volatile ether extract.

10 ALCOHOL EXTRACT[3].—OFFICIAL.

Place 2 grams of the sample in a 100 cc. flask and fill to the mark with 95 per cent alcohol by volume. Stopper, shake for 8 hours at 30 minute intervals and allow to stand for 16 hours longer without shaking. Filter the extract through a dry filter, evaporate a 50 cc. aliquot of the filtrate to dryness in a flat-bottomed dish on a water bath and heat to constant weight at 110°C.

11 COLD-WATER EXTRACT.—TENTATIVE.

(For ginger.)

Place 4 grams of the sample in a 200 cc. graduated flask, add water to the mark, shake at 30 minute intervals during 8 hours and let stand 16 hours longer without shaking. Filter and evaporate a 50 cc. aliquot of the filtrate to dryness in a flat-bottomed metal dish. Dry to constant weight at 100°C.

12 COPPER-REDUCING SUBSTANCES BY DIRECT INVERSION.—OFFICIAL.

Extract 4 grams of the sample with 5 successive portions of 10 cc. of ether on a filter that will retain completely the smallest starch granules. After the ether has evaporated, wash with 150 cc. of 10 per cent alcohol by volume.

Owing to the formation of a glutinous mass which clogs the filter, it is not possible to wash samples of Batavia cassia with water or dilute alcohol. Therefore all preliminary washing is best omitted in determinations made on all varieties of cassia, as well as on cassia buds and cinnamon.

Carefully wash the residue from the paper into a 500 cc. flask with 200 cc. of water, using a small wash bottle, and gently rubbing the paper with the tip of the finger. Hydrolyze and determine the copper reducing material as directed under **VII, 59**. Express the result in terms of starch.

13 STARCH.—OFFICIAL.

Extract 4 grams of the finely pulverized sample with ether and 10 per cent alcohol by volume, as directed under **12**, and determine starch by the diastase method, as directed under **VII, 61**.

14 CRUDE FIBER.—OFFICIAL.

Proceed as directed under **VII, 66**, and remove all ether extractives by successive washings of the dry fiber with ether previous to weighing.

15 TANNIN.—OFFICIAL.

(For cloves and allspice.)

Extract 2 grams of the sample for 20 hours with anhydrous ether. Boil the residue for 2 hours with 300 cc. of water, cool, make up to 500 cc. and filter. Measure 25 cc. of this infusion into a 1200 cc. flask, add 20 cc. of indigo solution, 750 cc. of water and proceed as directed under **XV, 30**. One cc. of N/10 oxalic acid is equivalent to 0.00623 gram of quercitannic acid, or 0.0008 gram of oxygen absorbed.

16 TOTAL SULPHUR.—OFFICIAL.

(For mustard.)

Proceed as directed under **II, 19**.

17 VOLATILE OIL IN MUSTARD SEED.—TENTATIVE.

Place 5 grams of the ground seed (No. 20 powder) in a 200 cc. flask, add 100 cc. of water, stopper tightly and macerate for 2 hours at about 37°C. Then add 20 cc. of 95 per cent alcohol by volume and distil about 60 cc. into a 100 cc. volumetric flask containing 10 cc. of 10 per cent ammonium hydroxid solution, taking care that the end of the condenser dips below the surface of the solution. Add 20 cc. of N/10 silver nitrate solution to the distillate, set aside overnight, heat to boiling on a water bath in order to agglomerate the silver sulphid, cool, make up to 100 cc. with water, and filter. Acidify 50 cc. of the filtrate with about 5 cc. of concentrated nitric acid and titrate with N/10 ammonium thiocyanate, using 5 cc. of 10 per cent ferric ammonium sulphate solution as an indicator. Each cc. of N/10 silver nitrate consumed equals 0.004956 gram of allylisothiocyanate.

OLIVE OIL.

(For paprika.)

18 *Qualitative Test.—Tentative.*

Spread 5 grams of the paprika on a watch glass and dry over sulphuric acid for at least 12 hours. Measure 250 cc. of anhydrous, alcohol-free ether [**VII, 9**] into a graduated flask on which the mark is situated near the lower end of the neck, and brush the paprika into it. Place a mark on the neck of the flask at the point where the meniscus is, and allow to stand for an hour, shaking at 20 minute intervals during that time. Bring the meniscus back to the mark placed upon the neck, either by cooling the flask and contents if the level has risen, or by adding absolute ether if it has fallen; let the solid particles settle and pipette off 100 cc. of the supernatant liquid, filter through an 11 cm. closely woven paper into a tared, air-dry, 250 cc., glass-stoppered Erlenmeyer flask that has been counterpoised against a similar flask; wash the paper with a little absolute ether. Then distil off the solvent and remove the flask from the bath as soon as the ether ceases to come over. Lay the flask on its side in a water oven and heat for 30 minutes, cool the open flask for at least 30 minutes in the air and weigh. Repeat this heating and weighing until the weight is constant to within 1 mg., two heatings usually being sufficient. Note the per cent of ether extract obtained. If more than 1.5 hours of heating are required to obtain constant weight, or if the ether extract becomes colorless, reject it and start a new determination with freshly purified ether.

Dissolve the ether extract in the flask with 10 cc. of chloroform, add 30 cc. of Hanus iodin solution [**XXII, 15 (a)**] and proceed as directed under **XXII, 16**, allowing 30 minutes for the halogen absorption. Note the iodin number of the ether extract. The iodin number of pure paprika thus obtained should not be less than 125.

MICROSCOPIC EXAMINATION.—TENTATIVE.

19 GENERAL.

Adulterants of vegetable origin in spices are best detected by means of the microscope. A general knowledge of vegetable histology and the microscopic appearance of the spices and spice adulterants is essential. Some of the standard works[4] on these subjects are listed in the bibliography.

20 REAGENTS.

Of the numerous reagents employed in histological work the following are the most useful in spice examinations:—

(a) *Glycerol solution (1 to 1).*

(b) *Absolute alcohol.*

(c) *Ether.*

(d) *Ammonium hydroxid.*—The concentrated solution, containing about 30 per cent of ammonia gas, is used in making Schweitzer's reagent and for some other purposes. For the tumeric test the concentrated solution should be diluted with 10 parts of water.

(e) *5 per cent potassium hydroxid solution.*

(f) *Chloral hydrate solution (8 to 5).*

(g) *Schultze's mixture.*—Crystallized potassium chlorate mixed with nitric acid as needed.

(h) *Iodin-potassium iodid solution.*—A solution of 0.05 gram of iodin, 0.2 gram of potassium iodid in 15 cc. of water.

(i) *Chlor-zinc iodin solution.*—Dissolve 100 grams of zinc chlorid in 60 cc. of water in a glass-stoppered bottle and add 20 grams of potassium iodid and 0.5 gram of iodin crystals. A few crystals of iodin should be left in the bottle to insure saturation and the solution should be allowed to stand a few hours before using. The chlor-zinc iodin solution, prepared in this manner, will keep for months. If the color developed in the tissue is too deep a blue, a very slight dilution of the reagent is advisable.

(j) *Millon's reagent.*—Prepare as directed under XIV, 9.

(k) *1 per cent ferric acetate or chlorid solution.*—Freshly prepared.

(l) *Alkanna tincture.*—Macerate 20 grams of alkanet root for several days with 100 cc. of alcohol.

(m) *Aqueous safranin solution.*

(n) *10 per cent hydrochloric acid.*

(o) *Acetic acid.*—Glacial or 99 per cent acetic acid diluted with 2 parts of water.

21 APPARATUS.

(a) *Dissecting microscope or hand lens.*

(b) *Compound microscope.*—Provided with $\frac{2}{3}$ and $\frac{1}{6}$ inch objectives, 1 and 2 inch oculars, double nosepiece, eyepiece micrometer and polarizing apparatus.

(c) *Sieves.*—A series of sieves with meshes ranging from 0.2 to 2 mm.

(d) *Slides, cover-glasses, needles, scalpels, forceps, etc.*

22 PREPARATION OF SAMPLE.

Reduce one portion to a fine powder in a mortar. Separate another portion into several grades of fineness by sieves of different mesh or by jarring on a sheet of paper. In the coarser grades, fragments of a suspicious nature may often be seen with the naked eye or under a simple microscope; these should be picked out for subsequent examination under the compound microscope.

23 EXAMINATION.

Mount a small quantity of the ground sample in water and examine under the compound microscope with both ordinary and polarized light. This gives general information as to the nature of the material and serves for the detection and identification of starch granules and various tissues. Draw a small drop of the iodin-potassium iodid solution into the same preparation by means of a piece of filter paper placed on the opposite edge of the cover-glass and examine. Starch granules will be colored blue or blue-black, cellulose yellow, and proteins either brown or yellow.

In the manner just described draw a little of the 5 per cent potassium hydroxid solution under the cover-glass and again examine. This treatment gelatinizes the starch granules, dissolves the proteins, saponifies the fats, and in other ways clears the preparation. It also imparts to tannins a reddish color. If this treatment does not clear the tissues satisfactorily, treat a fresh portion for some hours with the chloral hydrate solution.

Examine also the crude fiber obtained in the chemical analysis, as in this material the stone cells and other tissues are shown distinctly.

To isolate stone cells, bast fibers and other thick-walled cells macerate a portion of the sample in Schultze's mixture, using such proportion of potassium chlorate and nitric acid and heating for such a time as secures the desired results. Powdered charcoal and charred shells resist the bleaching action of potash, chloral hydrate and Schultze's mixture.

If it is desired to distinguish cellulose from infiltrated substances (lignin, suberin, etc.), add the freshly prepared chlor-zinc iodin solution to a water mount, whereby the former is colored blue and the latter yellow.

Test for proteins by cautiously warming on a slide with a drop of freshly prepared Millon's reagent. The proteins are partially decomposed, acquiring gradually a brick-red color. If it is desired to study the form of the aleurone (protein) granules, which in some plants are quite as characteristic as starch granules, prepare a mount in pure glycerol or oil.

To distinguish fats, oils, essential oils and resins from other cell contents, treat for an hour with alkanna tincture, diluted with an equal bulk of water, which imparts to these substances a deep red color, or treat with ether, which dissolves them. Treat also with alcohol, which dissolves the essential oils and resins, but does not perceptibly affect the fats and oils.

In testing for tannins and tissues impregnated with these substances, add the 1 per cent ferric acetate or chlorid solution. Both of these reagents give a green or blue color with tannins, but the former acts more slowly and is to be preferred.

Crystals of calcium oxalate are recognized by their characteristic forms and their behavior to polarized light. To distinguish calcium oxalate from calcium carbonate, treat with acetic acid, which does not affect the former, but dissolves the latter with effervescence. Both are soluble in hydrochloric acid.

PREPARED MUSTARD.

24 PREPARATION OF SAMPLE.—OFFICIAL.

Transfer the entire contents of the container to a dish sufficiently large to permit thorough stirring and make the whole mass homogeneous. Preserve in a bottle having a tightly fitting glass stopper. Stir well each time before removing a portion for analysis.

25

SOLIDS.—OFFICIAL.

Weigh 5 grams of the sample into a flat-bottomed, platinum dish, distribute evenly over the bottom of the dish with a little water, place on a water bath until the mixture appears dry, and heat finally to constant weight in a water oven.

26

ASH.—OFFICIAL.

Ignite the dry residue, obtained in the determination of solids, **24**, as directed under **VII, 4**.

27

SALT.—OFFICIAL.

Determine chlorin in the ash as directed under **II, 15** or **17**.

28

ETHER EXTRACT.—TENTATIVE.

Weigh 10 grams of the sample into a capsule and mix with about 30 grams of sand. Heat on a water bath until the mixture appears dry and complete the drying in a water oven. Grind until all the lumps are broken up, and determine the ether extract as directed under **VII, 10**.

29

PROTEIN.—OFFICIAL.

Determine the nitrogen as directed under **I, 18, 21** or **23**, using 5 grams of the sample. Multiply the result by 6.25 to obtain the equivalent amount of protein.

30

ACIDITY.—OFFICIAL.

Weigh 10 grams of the sample into a 200 cc. graduated flask, make up to the mark with water, shake, filter through a dry paper and determine the acidity in 100 cc. by titration with N/10 alkali, using phenolphthalein as an indicator. Express the result as acetic acid. One cc. of N/10 alkali is equivalent to 0.0060 gram of acetic acid.

31

COPPER-REDUCING SUBSTANCES.—OFFICIAL.

By Direct Inversion.

Proceed as directed under **VII, 59**, except that 10 grams of the sample, without previous washing or extraction, are treated directly with 200 cc. of water and 20 cc. of 25 per cent hydrochloric acid and the solution is made up to 250 cc. after neutralizing and before filtering and drawing off the aliquot. In analyses of samples containing starch, particular attention should be given that the amount of dextrose present in the aliquot taken for the reducing sugar determination does not exceed the maximum permitted for that determination. Express the result in terms of starch.

32

CRUDE FIBER.—TENTATIVE.

Transfer 8 grams of the sample (equivalent to about 2 grams of dry matter) to a porcelain or glass mortar. Treat with a little hot dilute sulphuric acid (1.25 grams per 100 cc.) and rub to a uniform thin paste. It is absolutely essential that this paste be uniform in consistency and entirely free from lumps. Rinse the thin mixture into a 500 cc. Erlenmeyer flask, using a total volume of 200 cc. of the hot dilute sulphuric acid for the entire operation. Proceed as directed under **VII, 66**, and remove all the fat, previous to weighing the crude fiber, by repeated washings of the dry fiber with ether.

33 COLORING MATTERS.—TENTATIVE.

Proceed as directed under X.

34 PRESERVATIVES.—OFFICIAL.

Proceed as directed under IX.

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XXIV. CACAO PRODUCTS.

1 PREPARATION OF SAMPLE.—OFFICIAL.

Mix powdered products thoroughly and preserve in tightly stoppered bottles. Chill sweet or bitter chocolate until it becomes hard and reduce to a finely granular condition by grating or shaving. Mix thoroughly and preserve in a tightly stoppered bottle in a cool place.

2 MOISTURE.—OFFICIAL.

Proceed as directed under VIII, 2.

3 ASH.—OFFICIAL.

Proceed as directed under VIII, 4, employing sufficient sample to contain approximately 1 gram of water-, sugar- and fat-free material.

4 ASH INSOLUBLE IN ACID.—OFFICIAL.

Proceed as directed under XXIII, 5.

5 SOLUBLE AND INSOLUBLE ASH.—OFFICIAL.

Proceed as directed under VIII, 14, employing sufficient sample to contain approximately 1 gram of water-, sugar- and fat-free material.

6 ALKALINITY OF THE SOLUBLE ASH.—OFFICIAL.

Proceed as directed under VIII, 15.

7 ALKALINITY OF THE INSOLUBLE ASH.—OFFICIAL.

Proceed as directed under VIII, 16.

8 TOTAL NITROGEN.—OFFICIAL.

Determine total nitrogen as directed under I, 18, 21 or 23.

9 CRUDE FIBER.—TENTATIVE.

Proceed as directed under VII, 66, employing sufficient sample to contain approximately 1 gram of water-, sugar- and fat-free material, except that both filtrations should be made upon paper, the washed fiber either being weighed upon a tared filter in the usual way or rinsed from the paper into a tared Gooch, dried and weighed.

The residue after fat extraction may be used directly for the crude fiber determination in the analysis of commercial cocoa and other finely ground or pulverized cacao products. If, however, the material is at all granular, it should be reduced to an impalpable powder, otherwise the results will be much too high. The pulverization may be satisfactorily performed by grinding with ether, as described under 10, treating the extracted residue with the hot dilute sulphuric acid and proceeding from this point as directed above.

STARCH.

10 *Direct Acid Hydrolysis Method.—Tentative.*

Weigh 4 grams of the sample, if unsweetened, or 10 grams if sweetened, into a small porcelain mortar, add 25 cc. of ether and grind. After the coarser material has settled, decant the ether, together with the fine suspended matter, on an 11 cm. paper of sufficiently fine texture to retain the crude starch. Repeat this treatment until no more coarse material remains. After the ether has evaporated from the filter, transfer the fat-free residue to the mortar by means of a jet of cold water and rub to an even paste, filtering on the paper previously employed. Repeat this process until all the sugar is removed. In the case of sweetened products the filtrate should measure at least 500 cc. Determine crude starch in the extracted residue as directed under **VII, 59.**

11 *Diastase Method.—Tentative.*

Remove fat and sugar from 4 grams of the sample, if unsweetened, or 10 grams if sweetened, as directed under **10**. Carefully wash the wet residue into a beaker with 100 cc. of water, heat to boiling over asbestos with constant stirring and continue the boiling and stirring for 30 minutes. Replace the water lost by evaporation and immerse the beaker in a water bath kept at 55°-60°C. When the liquid has cooled to the temperature of the bath, add 20 cc. of freshly prepared malt extract [**VII, 60**] and digest the mixture for 2 hours with occasional stirring. Boil a second time for 30 minutes, dilute, cool and digest as before with another 20 cc. portion of the malt extract. Heat again to boiling, cool and transfer to a 250 cc. flask. Add 3 cc. of alumina cream, make up to the mark and filter through a dry paper. The residue on the paper should show no signs of starch when examined microscopically. Continue from this point as directed under **VII, 61**, beginning with the words "Place 200 cc. of the filtrate in a flask with 20 cc. of hydrochloric acid".

12 FAT.—OFFICIAL.

Dry 2 grams of the material over sulphuric acid until all the moisture is practically removed. Products rich in fat show a tendency to cake at the temperature of boiling water. Hence, drying by means of heat must be avoided. Extract with anhydrous ether in a continuous extractor until no more fat is removed. Grind and repeat the extraction. A total extraction period of 4 hours is usually sufficient. Introduce the ether extract into a tared dish, allow the ether to evaporate and dry the residue to constant weight at 100°C.

The rapid centrifugal method[1], though useful and accurate under ordinary conditions, is unreliable during the summer months or in warm latitudes and has not been approved.

13 PHYSICAL AND CHEMICAL EXAMINATION OF THE FAT.

Separate the fat in a manner similar to that described under **15** and determine the melting point, index of refraction, iodin absorption, saponification, Reichert-Meissl and Polenske numbers as directed under **XXII**. Melting point determinations upon this material do not become normal until the fat has been kept for at least 24 hours in a cool place.

14 MILK FAT IN MILK CHOCOLATE.—TENTATIVE.

Estimate the amount of milk fat in milk chocolate from the following formula based on a Reichert-Meissl number of 0.5 for cocoa butter:

$$C = \frac{24A + 0.5B}{5} \text{ in which}$$

A = grams of butter fat in 5 grams of mixed fat,
B = 5 − A = grams of cocoa fat in 5 grams of mixed fat,
C = Reichert-Meissl number of extracted fat.

From which the

Weight of butter fat in 5 grams of mixed fat $= \frac{C - 0.5}{4.7}$ and the

Per cent of butter fat = per cent of total fat $\times \frac{C - 0.5}{23.5}$

15 SUCROSE AND LACTOSE.—TENTATIVE.

Prepare the sample by chilling well and shaving as finely as possible with a knife. Transfer 26 grams of this material to an 8 ounce nursing bottle, add about 100 cc. of petroleum ether and shake for 5 minutes. Centrifugalize until the solvent is clear. Draw off the clear solvent by suction and repeat the treatment with petroleum ether. Place the bottle containing the de-fatted residue in a warm place until the residual traces of petroleum ether are practically expelled. Add 100 cc. of water, shake until all the chocolate is loosened from the sides and bottom of the bottle and then shake for 3 minutes longer. Add basic lead acetate solution from a burette to complete precipitation, then sufficient water to make the total volume of added liquid 110 cc. Mix thoroughly and filter through a folded filter. Make the direct polariscopic reading "a" in a 200 mm. tube. Precipitate the excess of lead by anhydrous potassium oxalate and invert the solution as directed under VII, 14. Obtain the reading of the inverted solution. Multiply the invert reading by 2 to correct for dilution "b". From the figures obtained calculate the percentages of sucrose (S) and lactose (L) by the formulas

$$S = \frac{(a - b)\ (110 + x)}{142.66 - \frac{t}{2}}$$

$$L = \frac{a\left(1.1 + \frac{x}{100}\right) - S}{0.79} \quad \text{in which the value of x is obtained from}$$

$$x = \frac{0.2244\ (a - 21d)}{1 - 0.00204\ (a - 21d)} \quad \text{in which the value of d is obtained from}$$

$$d = \frac{a - b}{142.66 - \frac{t}{2}}$$

16 CASEIN IN MILK CHOCOLATE.—TENTATIVE.

It is unnecessary to de-fat the chocolate. Weigh 10 grams of the chocolate into a 500 cc. Erlenmeyer flask and add 250 cc. of 1 per cent sodium oxalate solution. Heat to boiling and boil gently for a few minutes, then cool, add 5 grams of magnesium carbonate and filter. Determine nitrogen in 50 cc. of this filtrate. Pipette 100 cc. of the filtrate into a 200 cc. volumetric flask and dilute almost to the mark with water. Then precipitate the casein by the addition of 2 cc. of glacial acetic acid or 1 cc. of concentrated sulphuric acid. Make to volume, shake, filter and determine nitrogen in 100 cc. of the filtrate. The difference between the two nitrogen determinations gives the nitrogen derived from the casein which, multiplied by 6.38, gives the amount of casein present in 2 grams of the sample.

17 COLORING MATTERS.—TENTATIVE.

Proceed as directed under X.

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XXV. COFFEES.

GREEN COFFEE.

1 MACROSCOPIC EXAMINATION.—TENTATIVE.

A macroscopic examination is usually sufficient to show the presence of excessive amounts of black and blighted coffee beans, coffee hulls, stones and other foreign matter. These can be separated by hand picking and determined gravimetrically.

2 COLORING MATTERS.—TENTATIVE.

Shake vigorously 100 grams or more of the sample with cold water or 70 per cent alcohol by volume. Strain through a coarse sieve and allow to settle. Identify soluble colors in the solution and insoluble pigments in the sediment as directed under X.

ROASTED COFFEE.

3 MACROSCOPIC EXAMINATION.—TENTATIVE.

Artificial coffee beans are apparent from their exact regularity of form. Roasted legumes and lumps of chicory, when present in whole roasted coffee, can be picked out and identified microscopically. In the case of ground coffee sprinkle some of the sample on cold water and stir lightly. Fragments of pure coffee, if not overroasted, will float, while fragments of chicory, legumes, cereals, etc., will sink immediately, chicory coloring the water a decided brown. In all cases identify the particles that sink by microscopical examination.

4 PREPARATION OF SAMPLE.—OFFICIAL.

Grind the sample to pass through a sieve having holes 0.5 mm. in diameter and preserve in a tightly stoppered bottle.

5 MOISTURE.—TENTATIVE.

Dry 5 grams of the sample at 105°–110°C. for 5 hours and subsequent periods of an hour each until constant weight is obtained. The same procedure may be used, drying in vacuo at the temperature of boiling water. In the case of whole coffee, grind rapidly to a coarse powder and weigh at once portions for the determination without sifting and without unnecessary exposure to the air.

6 SOLUBLE SOLIDS.—TENTATIVE.

Place 4 grams of the sample in a 200 cc. flask, add water to the mark and allow the mass to infuse 8 hours, with occasional shaking; let stand 16 hours longer without shaking, filter, evaporate 50 cc. of the filtrate to dryness in a flat-bottomed dish, dry at 100°C., cool and weigh.

7 ASH.—OFFICIAL.

Proceed as directed under VII, 4.

8 ASH INSOLUBLE IN ACID.—OFFICIAL.

Proceed as directed under XXIII, 5.

9 SOLUBLE AND INSOLUBLE ASH.—OFFICIAL.

Proceed as directed under VIII, 14.

10 ALKALINITY OF THE SOLUBLE ASH.—OFFICIAL.

Proceed as directed under VIII, 15.

11 SOLUBLE PHOSPHORIC ACID IN THE ASH.—OFFICIAL.

Acidify the solution of soluble ash, obtained in 9, with dilute nitric acid and determine phosphoric acid (P_2O_5) as directed under I, 6 or 9.

12 INSOLUBLE PHOSPHORIC ACID IN THE ASH.—OFFICIAL.

Determine phosphoric acid (P_2O_5) in the insoluble ash as directed under I, 6 or 9.

13 CHLORIDS.—OFFICIAL.

Proceed as directed under II, 20.

CAFFEIN.

14 *The Fendler and Stüber Method*[1].—*Tentative.*

Pulverize the coffee to pass without residue through a sieve having circular openings 1 mm. in diameter. Treat a 10 gram sample with 10 grams of 10 per cent ammonium hydroxid and 200 grams of chloroform in a glass-stoppered bottle and shake continuously by machine or hand for one-half hour. Pour the entire contents of the bottle on a 12.5 cm. folded filter, covering with a watch glass. Weigh 150 grams of the filtrate into a 250 cc. flask and evaporate on the steam bath, removing the last chloroform with a blast of air. Digest the residue with 80 cc. of hot water for 10 minutes on a steam bath, with frequent shaking, and let cool. Treat the solution with 20 cc. (for roasted coffee) or 10 cc. (for unroasted coffee) of 1 per cent potassium permanganate and let stand for 15 minutes at room temperature. Add 2 cc. of 3 per cent hydrogen peroxid (containing 1 cc. of glacial acetic acid in 100 cc.). If the liquid is still red or reddish, add hydrogen peroxid, 1 cc. at a time, until the excess of potassium permanganate is destroyed. Place the flask on a steam bath for 15 minutes, adding hydrogen peroxid in 0.5 cc. portions until the liquid becomes no lighter in color. Cool and filter into a separatory funnel, washing with cold water. Extract four times with 25 cc. of chloroform. Evaporate the chloroform extract from a weighed flask with aid of an air blast and dry at 100°C. to constant weight (one-half hour is usually sufficient). Weigh the residue as caffein and calculate on 7.5 grams of coffee. Test the purity of the residue by determining nitrogen and multiplying by 3.464 to obtain caffein.

15 *Modified Stahlschmidt Method*[2].—*Tentative.*

Weigh 3.125 grams of the finely powdered sample into a 500 cc. flask, add 225 cc. of water (this volume will be reduced to about 200 cc. by boiling), attach a reflux condenser and boil for 2 hours. Add 2 grams of dry basic lead acetate [VII, 13 (c)] and boil 10 minutes more. Cool, transfer to a 250 cc. graduated flask, fill to the mark, filter through a dry filter, measure 200 cc. of the filtrate into a 250 cc. graduated flask and pass hydrogen sulphid through it to remove the excess of lead. Make the solution up to the mark and filter through a dry filter. Measure 200 cc. of this filtrate into an evaporating dish and concentrate to about 40 cc. Wash the concentrated solution with as little water as possible into a small separatory funnel and shake out four times with chloroform, using 25, 20, 15 and 10 cc., respectively. If any emulsion forms, break it up with a stirring rod and run the separated portions of chloroform through a 5 cm. filter into a small, tared Erlenmeyer flask. Evaporate the chloroform on a steam bath, or recover the chloroform by attaching the flask to a condenser and distilling to a small volume. Dry the fine, white crystals of caffein to constant weight at 75°C. Test the purity of this residue by determining nitrogen as directed under **I, 18, 21** or **23** and multiplying by 3.464.

16 CRUDE FIBER.—OFFICIAL.

Proceed as directed under **VII, 66.**

17 STARCH.—TENTATIVE.

Extract 5 grams of the finely pulverized sample on a hardened filter with five successive portions (10 cc. each) of ether, wash with small portions of 95 per cent alcohol by volume until a total of 200 cc. have passed through, place the residue in a beaker and proceed as directed under **VII, 61.**

18 SUGARS.—TENTATIVE.

Proceed as directed under **VII, 56, 57** and **58.**

19 PETROLEUM ETHER EXTRACT.—OFFICIAL.

Dry 2 grams of the coffee at 100°C., extract with petroleum ether (b. p. 35°–50°C.) for 16 hours, evaporate the solvent, dry the residue at 100°C., cool and weigh.

20 TOTAL ACIDITY.—TENTATIVE.

Treat 10 grams of the sample, prepared as directed under **4**, with 75 cc. of 80 per cent alcohol by volume in an Erlenmeyer flask, stopper and allow to stand 16 hours, shaking occasionally. Filter and transfer an aliquot of the filtrate (25 cc. in the case of green coffee, 10 cc. in the case of roasted coffee) to a beaker, dilute to about 100 cc. with water and titrate with N/10 alkali, using phenolphthalein as an indicator. Express the result as the number of cc. of N/10 alkali required to neutralize the acidity of 100 grams of the sample.

21 VOLATILE ACIDITY.—TENTATIVE.

Into a volatile acid apparatus [**XV, 25,** Fig. 6] introduce a few glass beads and over these place 20 grams of the unground sample. Add 100 cc. of recently boiled water to the sample, place a sufficient quantity of recently boiled water in the outer flask and distil until the distillate is no longer acid to litmus paper. Usually 100 cc. of distillate will be collected. Titrate the distillate with N/10 alkali, using phenolphthalein as an indicator. Express the result as the number of cc. of N/10 alkali required to neutralize the acidity of 100 grams of the sample.

COATING AND GLAZING SUBSTANCES.

22 SUGAR AND DEXTRIN.—TENTATIVE.

Introduce 100 grams of the whole coffee into a beaker, add exactly 300 cc. of water, stir and allow to stand 5 minutes, with frequent stirring. Filter through a dry filter, add carefully to the filtrate sufficient dry lead acetate to precipitate all the caffetannic acid, avoiding an excess. Filter through a dry filter and remove the lead from the filtrate by the addition of a slight excess of anhydrous potassium oxalate. Filter through a dry filter and determine reducing sugars as invert sugar in 50 cc. of the filtrate, as directed under **VII, 25**. Invert a 75 cc. aliquot of the filtrate as directed under **VII, 14**. Cool, nearly neutralize with sodium hydroxid solution, make up to 100 cc. and determine reducing sugars as invert sugar in the resulting solution, as directed under **VII, 25**. Measure a 100 cc. aliquot of the filtrate into a 200 cc. flask, add 10 cc. of 25 per cent hydrochloric acid and hydrolyze as directed under **VII, 59**. Cool, neutralize with sodium hydroxid solution, make up to volume, filter through a dry filter and determine reducing sugars as invert sugar in 50 cc. of the filtrate, as directed under **VII, 25**. Calculate the reducing sugars in each instance to per cent by weight of the original coffee. Calculate sucrose from the reducing sugars before and after inversion as directed under **VII, 18**, and calculate dextrin as follows: Subtract the reducing sugars after inversion from the reducing sugars after hydrolysis, multiply the difference by the factor 0.9561 to convert the result to dextrose and then by 0.9 to convert to dextrin.

In some instances the presence of sucrose in the water extract may be verified by polarization. The presence of dextrin in the water extract may be verified by polarization as directed under **VIII, 22**, and by the erythro-dextrin test [**VIII, 44**] performed on the water extract previous to clarification with lead acetate.

23 EGG ALBUMEN AND GELATIN.—TENTATIVE.

Treat 100 grams of the whole coffee with 500 cc. of water and allow to stand with frequent stirring for 5 minutes. Filter and treat separate portions of the filtrate with (1) a strong solution of tannic acid, (2) Millon's reagent [**XIV, 9**]. Boil a third portion of the filtrate. In the presence of egg albumen a more or less heavy precipitate will be formed in each case. As a confirmatory test, treat an aliquot of the filtrate with an excess of tannic acid solution, add a little salt if necessary to secure flocculation of the precipitate, filter and, without washing, introduce the paper and its contents into a Kjeldahl flask and determine nitrogen. By this method coffee not coated with albumen or gelatin will yield less than 10 mg. of nitrogen per 100 grams of sample.

24 CHICORY INFUSION.—TENTATIVE.

Cover 100–150 grams of the whole coffee with water, allow to soak 2–3 minutes, stirring frequently, and drain the aqueous washings through a coarse sieve. Wash the coffee upon the sieve with about 100 cc. of water and centrifugalize the combined washings. Decant the clear liquid from the sediment, which should then be drained almost dry on filter paper. Mount the sediment in chloral hydrate [**XXIII, 20 (f)**] and examine under the microscope for elements of chicory.

25 FATS AND WAXES[3].—TENTATIVE.

Treat 100–200 grams of the beans with low boiling petroleum ether for 10 minutes, pour off the petroleum ether and repeat the process. Filter the combined extracts, evaporate and determine the index of refraction and the saponification number of the residue, as directed under **XXII, 6** and **20**.

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XXVI. TEAS.

1 DUST, STEMS AND FOREIGN LEAVES.—TENTATIVE.

Place 1 gram of the tea in a 300 cc. casserole, add 200 cc. of boiling water and allow to stand 15 minutes. This treatment will cause the leaves to unroll, so that they will be in condition for examination as to their form and structure[1]. A macroscopic examination will reveal the presence or absence of dust or stems.

2 PREPARATION OF SAMPLE.—OFFICIAL.

Grind the sample to pass through a sieve having circular openings 0.5 mm. in diameter.

3 MOISTURE.—OFFICIAL.

Proceed as directed under **VIII, 2.**

4 WATER EXTRACT[2].—TENTATIVE.

To 2 grams of the original sample in a 500 cc. Erlenmeyer flask add 200 cc. of hot water and boil over a low flame for an hour. The flask should be closed with a rubber stopper through which passes a glass tube 18 inches long for a condenser. The loss from evaporation should be replaced from time to time by the addition of hot water. Filter through a tared filter and wash the residue until the filtrate measures 500 cc., stirring the contents of the filter throughout the process to facilitate the filtering. Dry the filter paper and residue in the funnel in a steam oven until the excess of water is removed, transfer paper and contents to a tared weighing bottle and dry to constant weight at 100°C.

5 ASH.—OFFICIAL.

Proceed as directed under **VII, 4.**

6 SOLUBLE AND INSOLUBLE ASH.—OFFICIAL.

Proceed as directed under **VIII, 14.**

7 ASH INSOLUBLE IN ACID.—OFFICIAL.

Proceed as directed under **XXIII, 5.**

8 ALKALINITY OF THE ASH.—OFFICIAL.

Determine the alkalinity of the soluble and insoluble ash as directed under **VIII, 15** and **16.**

9 PHOSPHORIC ACID IN THE ASH.—OFFICIAL.

Determine phosphoric acid (P_2O_5) in the soluble and insoluble ash as directed under **XXV, 11** and **12.**

10 PETROLEUM ETHER EXTRACT.—OFFICIAL.

Proceed as directed under **XXV, 19.**

11 PROTEIN.—TENTATIVE.

Determine nitrogen as directed under **I, 18, 21** or **23.** Subtract the percentage of nitrogen present as caffein from the percentage of total nitrogen to obtain the percentage of nitrogen present as protein. Multiply this result by 6.25 to obtain the percentage of protein.

12 CRUDE FIBER.—OFFICIAL.

Proceed as directed under **VII, 66.**

13 VOLATILE OIL.—TENTATIVE.

Add 100 grams of tea to 800 cc. of water, distil, extract the distillate several times with petroleum ether, transfer the combined petroleum ether extracts to a tared dish, evaporate at room temperature, dry in a desiccator and weigh.

14 CAFFEIN.—TENTATIVE.

Proceed as directed under **XXV, 15.**

TANNIN[3].—TENTATIVE.

15 REAGENTS.

(**a**) *Potassium permanganate solution.*—Make up a solution containing 1.33 grams per liter and obtain its equivalent in terms of N/10 oxalic acid.

(**b**) *N/10 oxalic acid.*

(**c**) *Indigo carmine solution.*—Make up a solution containing 6 grams of indigo carmine (free from indigo blue) and 50 cc. of concentrated sulphuric acid per liter.

(**d**) *Gelatin solution.*—Soak 25 grams of gelatin for one hour in saturated sodium chlorid solution, heat until the gelatin is dissolved and make up to 1 liter after cooling.

(**e**) *Acid sodium chlorid solution.*—Acidify 975 cc. of saturated sodium chlorid solution with 25 cc. of concentrated sulphuric acid.

(**f**) *Powdered kaolin.*

16 DETERMINATION.

Boil 5 grams of the tea for 30 minutes with 400 cc. of water, cool, transfer to a 500 cc. graduated flask and make up to the mark. To 10 cc. of the infusion (filtered if not clear), add 25 cc. of the indigo carmine solution and about 750 cc. of water. Add from a burette the potassium permanganate solution, a little at a time while stirring, until the color becomes light green, then drop by drop, until the color changes to bright yellow or to a faint pink at the rim. Designate the number of cc. of permanganate used as "a".

Mix 100 cc. of the clear infusion of tea with 50 cc. of the gelatin solution, 100 cc. of the acid sodium chlorid solution and 10 grams of the powdered kaolin, and shake several minutes in a stoppered flask. After settling decant through a filter. Mix 25 cc. of the filtrate with 25 cc. of the indigo carmine solution and about 750 cc. of water and titrate with permanganate as before. The number of cc. of permanganate used subtracted from that obtained above, "a", gives the amount of permanganate required to oxidize the tannin. One cc. of N/10 oxalic acid is equivalent approximately to 0.0042 gram of tannin (gallotannic acid).

FACING.

17 GENERAL.—TENTATIVE.

Mineral pigments may be detected in the ash, or the tea may be shaken up with a large volume of water, and the water separated from the leaves by a sieve. The insoluble mineral substances used in facing will settle and can be removed by filtration for further examination, as directed under **X, 1**. The catechu and other soluble substances will be found in the filtrate.

18 PARAFFIN AND WAXY SUBSTANCES.—TENTATIVE.

Spread a quantity of the tea between two sheets of unglazed, white paper and place thereon a hot iron. Any greasy substance will stain the paper[4].

19 PIGMENTS USED FOR COLORING OR FACING[5].—TENTATIVE.

Place 60 grams of the tea in a 60 mesh, 5–6 inch sieve, provided with a top. Sift a small quantity (approximately 0.1 gram) of the dust upon a piece of semi-glazed, white paper about 8 by 10 inches. To obtain the requisite amount of dust, it is sometimes necessary to rub the leaf gently against the bottom of the sieve, but this must not be done until the sieve has been well shaken over the paper. Place the paper on a plain, firm surface, preferably glass or marble, and crush the dust by pressing firmly upon it a flat steel spatula about 5 inches long. Repeat the crushing process until the tea dust is ground almost to a powder when particles of coloring matter, if present, become visible as streaks on the paper. Brush off the loose dust and examine the paper by means of a simple lens magnifying 7.5 diameters. In distinguishing these particles and streaks bright light is essential. In many cases the character of the pigment is indicated by the behavior of these streaks when treated with reagents and examined under a microscope. The crushed particles of natural leaf in either black or green tea appear in such quantity that there is no chance of mistaking them for coloring or facing material. This test should be repeated, using black, semi-glazed paper for facings such as talc, gypsum, barium sulphate or clay.

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[5] Ibid.; Proc. Eighth Intern. Cong. Appl. Chem., 1912, 18: 301.

XXVII. BAKING POWDERS AND BAKING CHEMICALS.

1 PREPARATION OF SAMPLE.—OFFICIAL.

Remove the entire sample from the package, mix carefully and pass through a 20–40 mesh sieve.

TOTAL CARBON DIOXID.

2 *General Method.—Tentative.*

Make the determination by the absorption method, employing any apparatus which gives accurate results when checked with pure calcite. Whatever apparatus is chosen, the tubes and materials used for absorbing and drying the carbon dioxid may be varied according to the preference of the analyst. Use 0.25–1 gram of sodium or calcium carbonate, according to the amount of absorbent employed, and in the case of baking powder 0.50–2 grams.

Method Using Knorr's Apparatus.—Official.

3 REAGENTS.

(a) *Potassium hydroxid solution.*—Dissolve 25 grams of potassium hydroxid in 50 cc. of water.

(b) *Soda lime.*—Finely granulated and freed from dust by sifting.

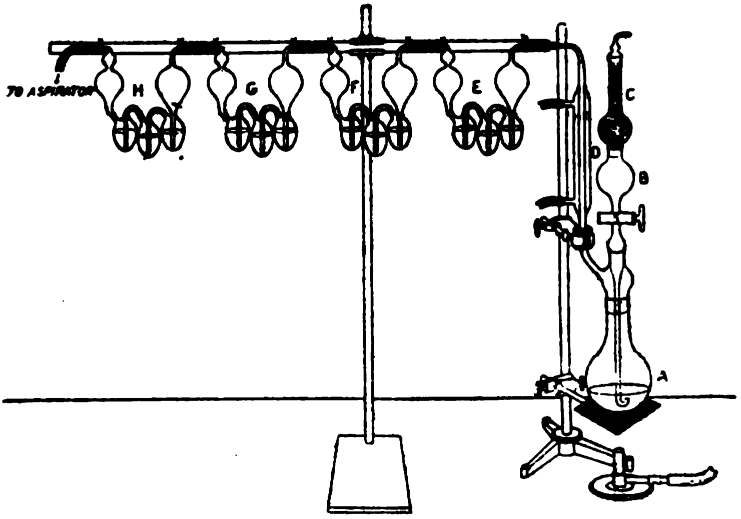

FIG. 12. KNORR'S APPARATUS FOR THE DETERMINATION OF CARBON DIOXID.

4 APPARATUS.

This consists of a flask (*A*), fitted by means of a ground-glass joint with a glass connection through the upper part of which passes a dropping funnel (*B*), and joined at the side with a Liebig condenser (*D*). The mouth of the dropping funnel (*B*) is connected by means of a perforated stopper with a soda lime tube (*C*). The upper end of the Liebig condenser is connected by a rubber joint with a Geissler bulb (*E*), containing sulphuric acid for drying the gas passing into the next Geissler bulb (*F*), connected with (*E*), and containing strong potassium hydroxid solution (1 to 2). The bulb (*F*) is connected with a third Geissler bulb (*G*), containing sulphuric acid for the absorption of moisture escaping from (*F*). A fourth Geissler bulb (*H*) is attached to (*G*) as a precaution to prevent moisture from the air being absorbed by (*G*). (*H*) is connected with an aspirator. Many analysts prefer to replace the bulb (*F*) by 2 U-tubes filled with sifted soda lime.

5 DETERMINATION.

Place 0.5–2 grams of the baking powder, the amount depending upon the percentage of carbon dioxid present, in the flask (*A*), which must be perfectly dry. Close the flask with the stopper which carries the funnel tube and the tube connecting with the absorption apparatus. Weigh separately the Geissler bulbs (*F*) and (*G*) and attach them to the apparatus. If 2 soda lime tubes are employed, weigh them separately and fill the first anew when the second increases materially in weight. Nearly fill the funnel tube (*B*) with hydrochloric acid (sp. gr. 1.1) and place the soda lime tube (*C*) in position. Then aspirate air through the Geissler bulbs at a rate of about 2 bubbles per second. Open the stopper of the funnel and allow the acid to run slowly into the flask, care being taken that the evolution of gas is so gradual as not to materially increase the current through the Geissler bulbs. After all the acid has been introduced, close the stop-cock in (*B*), continue the aspiration and heat gradually the contents of the flask to boiling. While the flask is being heated the aspirator tube may be removed, although many analysts prefer, when using ground-glass joints, to aspirate during the entire operation. Continue the boiling for a few minutes after the water has begun to condense in (*D*), then remove the flame, open the stop-cock in tube (*B*) and allow the apparatus to cool with continued aspiration. Remove the absorption bulbs (*F*) and (*G*) and weigh. The increase in weight is due to carbon dioxid.

Method Using Heidenhain's Apparatus.—Official.

6 REAGENTS.

(a) *Calcium chlorid.*—Use calcium chlorid dehydrated at 200°C., but not fused. Grind it coarsely in a coffee mill and sift through No. 18 wire gauze to remove the extremely coarse, and through No. 30 wire gauze to remove the very fine, particles.

(b) *Soda lime.*—Grind and sift the soda lime[1] for the weighed tubes as described above. It should not be too dry, as it must not absorb moisture to a greater degree than the calcium chlorid.

7 APPARATUS[2].

This consists of a cylinder (*A*), filled with soda lime to remove carbon dioxid from the air passing through the apparatus. A thick layer of cotton at the upper end prevents soda lime dust from being carried over. Connect the cylinder (*A*) by means of a perforated rubber stopper and a bent glass tube having a stop-cock (*B*) and a capillary constriction (*C*) with a short piece of rubber tubing to which is attached a short piece of glass tubing (*E*), fitted with a perforated rubber stopper. The latter fits tightly into the constriction of the funnel tube (*D*). The funnel of the latter is cylindrical in shape, ¾ inch in diameter at the upper end, ⅜ inch at the lower end and 4 inches long, the rubber stopper of (*E*) fitting into the constriction. The stem of the funnel tube (*D*) passes through a doubly perforated rubber stopper almost to the bottom of the evolution flask (*F*), which is ordinarily of 150 cc. capacity but, in the case of foaming liquids, may hold 300 cc. Through the second perforation in the stopper connect the evolution flask (*F*) with a reflux condenser (*G*), consisting of a ¼ inch glass tube around which is wound a small lead pipe carrying a current of cold water. To the upper end of the condenser attach a U-tube containing a little calcium chlorid (to be renewed when it has liquefied) to retain the bulk of the moisture. Connect this U-tube with a second U-tube (*H*), filled with coarse calcium chlorid, and this in turn with a third U-tube (*K*), filled at (*I*) with a 3 inch column of pumice stone impregnated with copper sulphate and completely dehydrated at 150°C., the remainder of the tube being filled with fine calcium chlorid. Connect the U-tube (*K*) with a bent glass tube having a stop-cock (*L*) which is closed when the apparatus is not in use. Next attach the absorption U-tubes (*M*) and (*N*) which are ½ inch in diameter and 5 inches long, the first filled mainly with soda lime but containing a little calcium chlorid at the end where the air current enters, the second filled one-half with soda lime and one-half with calcium chlorid, the latter being placed at the side where the air current leaves. Connect (*N*) with a guard tube (*O*), filled with calcium chlorid on the side toward (*N*) and with soda lime on the side toward (*P*), the latter being a small U-tube trapped with glycerol to indicate the passage of the air current. Connect (*P*) with a safety bottle (*R*), to receive any water which may be sucked back from the aspirator, and connect (*R*) with the aspirator (*S*), a 4 liter Mariette bottle.

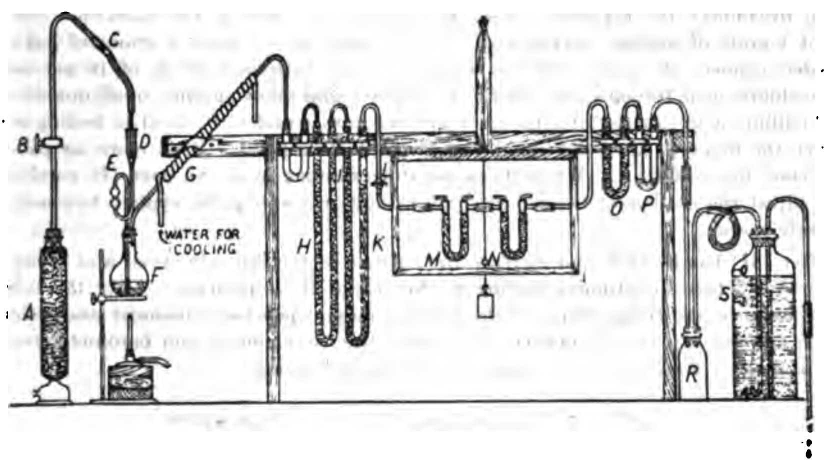

FIG. 13. HEIDENHAIN'S APPARATUS FOR THE DETERMINATION OF CARBON DIOXID.

The tubes (*M*) and (*N*) should hold about 20 grams, making the capacity of (*M*) for carbon dioxid almost 1 gram and that of (*N*) for moisture 0.2 gram. (*M*) should be refilled when its weight has increased 0.75 gram and (*N*) after increase of 0.1 gram in weight.

Use the best grade of rubber for all connections, applying a trace of castor oil as a lubricant. For connections of the weighed tubes use rubber tubing boiled in weak lye, washed and dried. Apply also a little castor oil, which is thoroughly wiped off again before connecting the tubing.

Before using the apparatus fill (*H*) and (*K*) with carbon dioxid in order to saturate the alkalinity of the calcium chlorid and exhaust after several hours.

8 DETERMINATION.

In order to find the allowable rapidity of the air current employed during the determination, proceed as follows: Charge the apparatus exactly as for an analysis, leaving out the carbonate. Begin to aspirate at the rate of about 50 cc. per minute. After 2 liters have been aspirated weigh the tubes, (*M*) and (*N*). If they have lost weight, repeat the experiment with 40 cc. per minute, and so on until the weight of the tubes remains constant. If the work has been done with due precaution, the first tube should have lost just as much as the second has gained. Do not exceed the safe speed thus found.

Weigh the tubes (*M*) and (*N*) at the air temperature of the balance room. Shortly before weighing open the tubes for a moment to allow equalization of air. Note the thermometer and barometer readings. Connect the tubes with the apparatus and test the tightness of the joints by closing (*A*) at the bottom, opening all the cocks, starting the aspirator, and observing (*P*), in which the liquid should soon come to a standstill. Then disconnect the aspirator, close (*B*), remove (*F*), put in the substance, using about 1 gram of sodium carbonate or calcium carbonate or about 2 grams of baking powder, connect (*F*), and start the condenser (*G*). Introduce 50 cc. of 10 per cent hydrochloric acid through (*D*), lifting (*E*) slightly and allowing only small quantities of the dilute acid to enter at a time. Light the burner under (*F*), heat to boiling and reduce the flame to keep the liquid just at the boiling point. If no more air passes (*P*), start the aspiration. When the water stops running from (*S*), open (*B*) carefully and adjust the outflow of the aspirator by raising or lowering the syphon to one-half the safe speed.

After (*M*) has become cool increase the current to the full safe speed and aspirate altogether 3 liters, continuing boiling to the end of the aspiration. After the tubes have assumed the temperature of the balance room, open for a moment and weigh. When extreme accuracy is desired, note again the thermometer and barometer readings and apply correction according to the following formula:

$$-(A^2 - A^1) \times T \text{ and } + (B^2 - B^1) \times B \text{ in which}$$

A^1 = the temperature at first weighing in degrees C.;
A^2 = the temperature at second weighing in degrees C.;
B^1 = the barometric pressure at first weighing in mm.;
B^2 = the barometric pressure at second weighing in mm.;

T and B are constants found from the following formulas:

$$T = V \times 0.0000039 \text{ gram};$$
$$B = V \times 0.0000015 \text{ gram in which}$$

0.0000039 = change in weight of 1 cc. of air for 1°C.;
0.0000015 = change in weight of 1 cc. of air for 1 mm. pressure;

and the value of V is obtained from

$$V = \frac{G}{2.7} + \frac{F}{2.0} - \frac{G+F}{8.5},$$

representing the differential volume affected by temperature and pressure and being a constant for the tubes and in which

G = the weight of the empty tubes;
F = the weight of the fillings;
2.7 = the specific gravity of glass;
2.0 = the specific gravity of filling;
8.5 = the specific gravity of brass;

$$\frac{G}{2.7} + \frac{F}{2.0} = \text{volume of tubes and fillings;}$$

$$\frac{G+F}{8.5} = \text{volume of brass weights.}$$

9 RESIDUAL CARBON DIOXID[3].—OFFICIAL.

Weigh 2 grams of the baking powder into a flask suitable for the subsequent determination of carbon dioxid, add 20 cc. of cold water and allow to stand 20 minutes. Place the flask in a metal drying cell surrounded by boiling water and heat, with occasional shaking, for 20 minutes.

To complete the reaction and drive off the last traces of gas from the semi-solid mass, heat quickly to boiling and boil for a minute. Aspirate until the air in the flask is thoroughly changed, and determine the residual carbon dioxid by absorption, as directed under **5** or **8**.

The process described[3], based on the methods of McGill[4] and Catlin[5], imitates as far as practicable the conditions encountered in baking, but in such a manner that concordant results may be readily obtained on the same sample and comparable results on different samples.

10 AVAILABLE CARBON DIOXID.—OFFICIAL.

Subtract the residual carbon dioxid from the total.

11 ACIDITY.—OFFICIAL.

(For cream of tartar and its substitutes.)

Dissolve 1 gram of the sample in hot water and titrate with N/5 potassium hydroxid, using phenolphthalein as an indicator.

12 TARTARIC ACID, FREE OR COMBINED[6].—TENTATIVE.

(Applicable in the presence of phosphates.)

Shake repeatedly about 5 grams of the sample with about 250 cc. of cold water in a flask and allow the insoluble portion to subside. Decant the solution through a filter and evaporate the filtrate to dryness. Powder the residue, add a few drops of 1 per cent resorcin solution and about 3 cc. of strong sulphuric acid and heat slowly. Tartaric acid is indicated by a rose-red color which is discharged on dilution with water.

13 TOTAL TARTARIC ACID.—OFFICIAL.

(Applicable only in the absence of aluminium salts, calcium salts and phosphates.)

Into a shallow 6 inch porcelain dish weigh out 2 grams of the sample and sufficient potassium carbonate to combine with all the tartaric acid not in the form of potassium bitartrate. Mix thoroughly with 15 cc. of cold water and add 5 cc. of 99 per cent acetic acid. Stir for 30 seconds with a glass rod bent near the end. Add 100 cc. of 95 per cent alcohol by volume, stir violently for 5 minutes, and allow to settle at least

30 minutes. Filter on a Gooch crucible with a thin layer of paper pulp and wash with 95 per cent alcohol by volume until 2 cc. of the filtrate do not change the color of litmus tincture diluted with water. Place the precipitate in a small casserole, dissolve in 50 cc. of hot water and add N/5 potassium hydroxid, leaving it still strongly acid. Boil for a minute. Finish the titration, using phenolphthalein as an indicator and correct the reading by adding 0.2 cc. One cc. of N/5 potassium hydroxid, under the above conditions, is equivalent to 0.02641 gram of tartaric anhydrid, 0.03001 gram of tartaric acid, or 0.03763 gram of potassium bitartrate. Standardize the N/5 potassium hydroxid by means of pure potassium bitartrate.

The accuracy of this method is indicated by the agreement of the percentages of potassium bitartrate in cream of tartar powders containing no free tartaric acid, obtained by calculation from the tartaric acid, with those obtained by calculation from the potassium oxid[7].

FREE TARTARIC ACID.

14 *Qualitative Test.—Official.*

Extract 5 grams of the sample with absolute alcohol and evaporate the alcohol from the extract. Dissolve the residue in dilute ammonium hydroxid, transfer to a test tube, add a good sized crystal of silver nitrate and heat gently. Tartaric acid is indicated by the formation of a silver mirror. If desired, the absolute alcohol extract may be tested as directed under **12**.

15 *Quantitative Method.—Official.*

Calculate the percentage of tartaric anhydrid combined with the potash as bitartrate, if any, and subtract this from the percentage of total tartaric anhydrid. The difference is the tartaric anhydrid originally added as the free acid, although, if the sample has been kept for a long time or has been improperly stored, a portion or all of this acid may exist at the time of analysis as the sodium salt resulting from the reaction in the can with the sodium bicarbonate. Multiply by 1.137 to obtain the percentage of tartaric acid.

16 POTASSIUM BITARTRATE.—OFFICIAL.

If, as is usually the case, potassium bitartrate is the only potassium salt present, multiply the percentage of total potash, determined as directed under **24**, by 3.994.

STARCH.

17 *Direct Inversion Method.—Official.*

(For all baking powder ingredients free from lime.)

Weigh 5 grams of the powder into a 500 cc. graduated flask and proceed as directed under **VII, 59**.

18 *Indirect Method[7].—Official.*

(For phosphate, alum phosphate and all other baking powders containing lime.)

Mix 5 grams of the powder with 200 cc. of 3 per cent hydrochloric acid in a 500 cc. graduated flask and allow the mixture to stand for an hour, with frequent shaking. Filter on an 11 cm. hardened filter, taking care that a clear filtrate is obtained. Rinse the flask once without attempting to remove all the starch, and wash the paper twice with cold water. Carefully wash the starch from the paper back into the flask with 200 cc. of water. Add 20 cc. of 25 per cent hydrochloric acid and proceed as directed under VII, 59.

The treatment with 3 per cent hydrochloric acid, without dissolving the starch, removes effectively the lime, which otherwise would be precipitated as tartrate by the alkaline copper solution.

19 *Modified McGill Method.—Tentative.*

Digest 1 gram of the powder with 150 cc. of 3 per cent hydrochloric acid for 24 hours at room temperature, with occasional shaking. Filter on a Gooch crucible, wash thoroughly with cold water and then once each, with alcohol and ether. Dry at 110°C. (4 hours is usually sufficient), cool and weigh. Burn off the starch, weigh again and determine the starch by difference.

The results by this method on cream of tartar powders and tartaric acid powders agree closely with those obtained by copper reduction. On phosphate, alum and alum-phosphate powders the results are usually satisfactory, but in some instances they may be over 2 per cent too high.

ALUM IN THE PRESENCE OF PHOSPHATES[9].

20 *Qualitative Test.—Official.*

(**a**) *In baking powder.*—Burn about 2 grams of the sample to an ash in a porcelain dish. Extract with boiling water and filter. Add to the filtrate a few drops of ammonium chlorid solution. A flocculent precipitate indicates alum.

(**b**) *In cream of tartar.*—Mix about 1 gram of the sample with an equal quantity of sodium carbonate, burn to an ash and proceed as in (**a**).

ASH[10].

21 INSOLUBLE ASH AND PREPARATION OF SOLUTION.—OFFICIAL.

Char 5 grams of the sample in a platinum dish at a heat below redness. Boil the carbonaceous mass with dilute hydrochloric acid, filter into a 500 cc. graduated flask and wash with hot water. Return the residue, together with the paper, to the platinum dish and burn to a white ash. Boil again with hydrochloric acid, filter, wash, unite the 2 filtrates and dilute to 500 cc.

Incinerate the residue after the last filtration and determine the ash insoluble in acid.

22 IRON AND ALUMINIUM.—OFFICIAL.

Draw a 100 cc. aliquot of the solution, prepared as directed under **21**, and separate silica, if necessary. Mix the solution with sodium phosphate solution in excess. Add ammonium hydroxid until a permanent precipitate is obtained, then hydrochloric acid, drop by drop, until the precipitate is dissolved. Heat the solution to about 50°C., mix with a considerable excess of 50 per cent ammonium acetate solution and 4 cc. of 80 per cent acetic acid.

As soon as the precipitate of aluminium phosphate, mixed with iron phosphate, has settled, collect on a filter, wash with hot water, ignite and weigh.

Fuse the mixed phosphates with 10 parts of sodium carbonate, dissolve in dilute sulphuric acid, reduce with zinc and determine the iron by titration with a standard permanganate solution. In the same solution determine the phosphoric acid, as directed under **I, 6** or **9**. To obtain the weight of alumina (Al_2O_3), subtract the sum of the weights of ferric oxid (Fe_2O_3) and phosphorus pentoxid (P_2O_5) from the weight of the mixed phosphates.

23 CALCIUM.—OFFICIAL.

Heat the combined filtrate and washings, obtained as directed under **22**, to 50°C and add an excess of ammonium oxalate solution. Allow to stand in a warm place until the precipitate has settled, filter, wash the precipitate with hot water, dry, ignite over a Bunsen burner and finally over a blast lamp. Cool in a desiccator and weigh as calcium oxid.

24 POTASSIUM AND SODIUM.—OFFICIAL.

Evaporate an aliquot of the solution, prepared as directed under **21**, nearly to dryness to remove the excess of hydrochloric acid, dilute and heat to boiling. While still boiling add barium chlorid solution as long as a precipitate forms and then enough barium hydroxid solution to make the liquid strongly alkaline. As soon as the precipitate has settled, filter and wash with hot water, heat the filtrate to boiling, add sufficient ammonium carbonate solution (1 part of ammonium carbonate in 5 of 2 per cent ammonium hydroxid solution) to precipitate all the barium, filter and wash with hot water. Evaporate the filtrate to dryness and ignite the residue below redness to remove ammonium salts. Add to the residue a little water and a few drops of ammonium carbonate solution. Filter into a tared platinum dish, evaporate, ignite below redness and weigh the mixed potassium and sodium chlorids.

Determine potassium in the mixed chlorids as directed under **I, 45**, beginning with "Digest the residue with hot water, filter through a small filter". Calculate the potassium so found to its equivalent of potassium chlorid and subtract this from the weight of the mixed chlorids to obtain the weight of sodium chlorid.

25 PHOSPHORIC ACID.—OFFICIAL.

Mix 5 grams of the sample with a little magnesium nitrate solution, dry, ignite, dissolve in dilute hydrochloric acid and dilute the solution to a definite volume. In an aliquot of the solution determine phosphoric acid as directed under **I, 6** or **9**.

26 SULPHURIC ACID[11].—OFFICIAL.

Boil 5 grams of the sample gently for 1.5 hours with a mixture of 300 cc. of water and 15 cc. of concentrated hydrochloric acid. Dilute to 500 cc., draw off a 100 cc. aliquot, dilute considerably, precipitate with 10 per cent barium chlorid solution, filter the precipitated barium sulphate on a Gooch, wash with hot water, dry, ignite and weigh.

27 AMMONIA.—OFFICIAL.

Introduce 2 grams of the sample into a distillation flask, add 300–400 cc. of water and an excess of sodium hydroxid solution, connect with a condenser and distil into a measured amount of standard acid. Titrate the excess of acid in the distillate with standard alkali, using methyl red or cochineal as an indicator.

Ammonia alum is often an ingredient of cream of tartar substitutes and baking powders, and ammonium carbonate is occasionally present in baking powders.

LEAD.

Method I. Colorimetric Method[12].—*Tentative.*

(Applicable in the absence of alum and phosphates. Approximate method for preliminary work.)

28 REAGENTS.

(Lead-free reagents must be used throughout all operations.)

(**a**) *Sodium bisulphite solution.*—Dissolve 10 grams of anhydrous sodium carbonate in sufficient water to make 100 cc. and pass sulphur dioxid into the solution until carbon dioxid is no longer evolved. Dilute a little of this solution with 10 volumes of water as needed in the determination.

(**b**) *Potassium cyanid solution.*—Dissolve 10 grams of the salt in sufficient water to make 100 cc.

(**c**) *Standard lead solution.*—Dissolve 1.6 grams of crystallized lead nitrate, previously dried over sulphuric acid, in a liter of water containing a few drops of dilute nitric acid. One cc. of this solution is equivalent to 1 mg. of metallic lead. Dilute 1 cc. of this solution to 100 cc. immediately before use in making up the color standards.

(**d**) *Lead-free tartrate solution.*—Dissolve 200 grams of tartaric acid in about 500 cc. of hot water, cool, add 40 cc. of the sodium bisulphite solution, heat to incipient boiling and test a few drops of the solution with potassium thiocyanate solution to ascertain if all the iron is reduced to the ferrous state, repeating the treatment with about 10 cc. of the sodium bisulphite solution in case ferric iron is still present. Cool, add 20 cc. of the 10 per cent potassium cyanid solution, and then strong ammonium hydroxid solution until the solution is distinctly alkaline to litmus paper. Boil until the solution is clear, cool, add 2 cc. of freshly prepared, colorless ammonium sulphid solution, dilute to 1 liter and allow to stand overnight. Filter to remove the precipitated sulphids, boil the filtrate until hydrogen sulphid is removed, cool and dilute to 1 liter with water.

29 PREPARATION OF SOLUTION.

(**a**) *Baking powder.*—Weigh 20 grams of the sample into a 250 cc. casserole, add water a little at a time with stirring until foaming ceases, then hydrochloric acid (1 to 1) a little at a time until all the carbonate is decomposed and finally 5 cc. excess of the hydrochloric acid. Cover with a watch glass and digest on a steam bath until all the starch is hydrolysed as shown by testing 1 or 2 drops of the mixture with iodin. Filter through a folded filter and wash the filter several times with small portions of hot water. Treat the residue on the filter with several small portions of hot nitric acid (sp. gr. 1.2), collect the acid solution in a separate, small porcelain dish, evaporate this solution to dryness on a water bath and expel nitric acid by several treatments and evaporations with a few drops of concentrated hydrochloric acid. Rinse the contents of the dish through a small filter into the main solution and make up to 100 cc.

(**b**) *Tartaric acid and cream of tartar.*—Dissolve 100 grams of the sample in hot water, add 50 cc. of hydrochloric acid (1 to 1), filter into a liter graduated flask and wash the filter several times with water. Then treat the residue on the filter with several small portions of hot nitric acid (sp. gr. 1.2), collect the acid solution in a separate, small porcelain dish, evaporate this solution to dryness on a water bath and expel nitric acid by several treatments and evaporations with a few drops of concentrated hydrochloric acid. Rinse the contents of the dish through a small filter into the main solution, finally diluting the combined filtrates and washings to 1 liter.

30 DETERMINATION.

Introduce 50 cc. of the solution, prepared as directed under **29**, into a beaker, add 2 cc. of the sodium bisulphite solution, heat to incipient boiling, and test a few drops of the solution with potassium thiocyanate to determine if all the iron is reduced to the ferrous state, repeating the treatment with the sodium bisulphite solution if ferric iron is still present. Cool, add 1 cc. of the 10 per cent potassium cyanid solution and neutralize to litmus with strong ammonium hydroxid solution; finally add an excess of 1 cc. of the last reagent. Boil gently until clear and colorless, cool and make up to 100 cc. Treat with 2 drops of freshly prepared, colorless ammonium sulphid solution, mix and compare in a colorimeter with standard solutions, prepared by adding measured amounts of the standard lead solution to 50 cc. of the lead-free tartrate solution, diluting to 100 cc. and treating with 2 drops of freshly prepared colorless ammonium sulphid solution.

The final comparison should be made with a standard containing approximately the same amount of lead, and the addition of ammonium sulphid solution should be made to the standards and the solution of the sample at the same time, as the colors change on standing.

31 *Method II.—Tentative.*

(Applicable to alum or phosphate baking powders or their ingredients.)

Weigh 100 grams of the sample into a 1.3 liter beaker and add an excess of hydrochloric acid (1 to 3) in small portions, keeping down excessive frothing with a little ether. Heat the mixture on a steam bath until the starch is hydrolyzed and the solution is quite limpid. Cool and add 200 cc. of 50 per cent lead-free ammonium citrate solution. Place the beaker in a bath of cold water and add carefully ammonium hydroxid solution, in small portions with constant stirring, until the mixture is alkaline. If a precipitate forms, add sufficient ammonium citrate solution to dissolve it. Then add 15 cc. of saturated mercuric chlorid solution, dilute the mixture to about 1200 cc., saturate with hydrogen sulphid, and allow to stand until the precipitate has settled (15–20 minutes). Filter and wash the precipitate with hydrogen sulphid water. Place the paper and precipitate in a small casserole, add 10 cc. of concentrated nitric acid and 2 cc. of concentrated sulphuric acid and heat on a hot plate until the mixed acids have been slowly driven off. Heat the residue in a muffle at low redness until the mercury salts have volatilized. Cool the casserole and leach the residue several times with 25 per cent ammonium acetate solution, made slightly alkaline with ammonium hydroxid, pass the leachings through a small filter into a beaker and finally wash the residue and filter paper with a little hot water. Acidify the combined filtrate and washings with acetic acid, add an excess of potassium dichromate solution and allow to stand overnight. Filter on a tared Gooch, wash with water, dry for 30 minutes at 125°–150°C., cool and weigh as lead chromate. Calculate the weight of metallic lead, using the factor 0.641. Conduct a blank determination upon all the reagents and correct the result accordingly.

32 *Method III.—Tentative.*

(Applicable in the absence of alum and phosphates.)

Weigh 100 grams of the sample into a liter beaker and add an excess of hydrochloric acid (1 to 3) in small portions, keeping down excessive frothing with a little ether. Heat the mixture on a steam bath until the starch is hydrolyzed and the solution is quite limpid. Cool, add ammonium hydroxid solution until distinctly alkaline, dilute to about 800–900 cc. and saturate with hydrogen sulphid. Allow the mixture to stand for 3–4 hours or until the precipitate has settled, filter on a 12.5 cm. close-textured paper and wash the precipitate several times with hydrogen sulphid water. Place the filter paper and precipitate in a 100 cc. Erlenmeyer flask, add 5 cc. of concentrated sulphuric acid and 5 cc. of concentrated nitric acid and heat on a hot plate, with occasional additions of small portions of concentrated nitric acid, until the mixture no longer blackens when evaporated to the point at which white fumes of sulphur trioxid appear. Cool, dilute with 20 cc. of water, warm until the ferric sulphate goes into solution, cool and then add 40 cc. of 95 per cent alcohol by volume. Allow to stand overnight, filter on a Gooch and wash with 95 per cent alcohol. Dissolve the lead sulphate remaining on the filter by washing with 20 cc. of 25 per cent ammonium acetate solution, rendered slightly alkaline with ammonium hydroxid, collect the filtrate in a small beaker, passing it through the filter 3 or 4 times. Finally wash the filter with hot water, acidify the combined filtrate and washings with acetic acid, add an excess of potassium dichromate solution and allow to stand overnight. Filter on a small, tared Gooch, wash, dry for 30 minutes at 125°–150°C. and weigh as lead chromate. Calculate the metallic lead, using the factor 0.641.

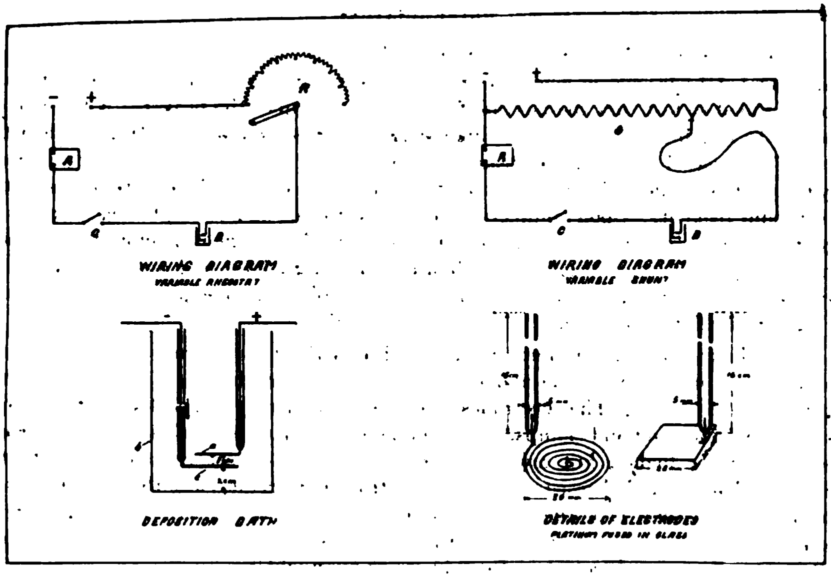

FIG. 14. APPARATUS FOR ELECTROLYTIC DETERMINATION OF LEAD.

Wiring Diagrams:
A = ammeter.
B = deposition bath.
C = switch.
R = variable rheostat.
S = variable shunt.

Deposition Bath:
a = anode (plate).
b = beaker.
c = cathode (spiral).

Details of Electrodes:
Spiral = 30 cm. of No. 22 wire.
Plate = foil—25 mm. square.

Electrolytic Method.—Tentative.

33

APPARATUS.

(a) *Anode.*—Weld a piece of platinum wire 1–2 cm. long to a piece of platinum foil 25 mm. square, the free end of the wire being fused into a glass tube as shown in Fig. 14.

By partly filling the tube with mercury and inserting the wire from the circuit, contact is made with the anode. Test the platinum-glass joint to make sure that no mercury can leak through.

(b) *Cathode.*—Fuse one end of a piece of No. 22 platinum wire 30 cm. long into a glass tube in the same manner as described in (a), and wind the wire into a flat coil 30 mm. in diameter, the plane of the coil to be perpendicular to the axis of the glass tube as shown in Fig. 14.

(c) *A source of direct current* with a potential of at least 6 volts, which may be varied by means of a rheostat or shunt resistance, as indicated in Fig. 14.

(d) *A D C ammeter* graduated at least to 0.1 ampere divisions and having a range of 0–5 amperes. Place in circuit as shown in Fig. 14.

34 DETERMINATION.

Place 100 grams of baking powder in a tall 500 cc. beaker. Slowly add 75 cc. of concentrated hydrochloric acid (sp. gr. 1.18), stirring continuously. The acid must not be added so fast that the mixture foams over the top of the beaker. A thick paste results, to which add 75–100 cc. of luke warm water, slowly with stirring. When the action has ceased, place on a steam bath and warm until the starch is completely hydrolyzed, as indicated by a negative reaction when a drop of the mixture is tested with iodin reagent. Upon completion of the hydrolysis add concentrated ammonium hydroxid carefully from a burette until the first trace of permanent precipitate is formed, then add 5 cc. of concentrated hydrochloric acid (sp. gr. 1.18) and make up to 400 cc. with water. Warm if necessary to completely dissolve the precipitate. The mixture is then ready for electrolysis.

Insert the cathode in the solution till the spiral is about 2 cm. from the bottom of the beaker, then insert the anode above the cathode so that there is 1 cm. between the spiral and the plate. The electrodes may be conveniently supported in the proper position by ringstand and clamps. The temperature during electrolysis should be room temperature or higher, but must not exceed 50°C. Connect the electrodes to the circuit and with the full resistance in the electrolyzing circuit, close the switch. (With the variable rheostat type the total resistance must be large enough to cut the current down to approximately 0.1 ampere.) Adjust the resistance so that the ammeter shows 0.3 ampere flowing. Electrolyze for 8 hours or overnight[18].

Upon completion of the electrolysis, considerable quantities of salts will frequently be found adhering to the lead deposit on the cathode. Carefully remove the beaker without jarring the electrodes, while the current is still on. Replace this beaker immediately by one of the same size, full of water to which 2 cc. of concentrated hydrochloric acid has been added. Allow the current to run until the salts have been removed from the electrodes, then without interrupting the current replace the beaker with one full of water and continue the current for 30 minutes.

Remove the beaker of water, open the switch, and wash the cathode in a 50 cc. beaker containing sufficient alcohol to cover the lead deposit. Dry in air, and dissolve the lead from the cathode with 3–4 cc. of concentrated nitric acid in a 50 cc. beaker, heating gently to aid solution. Wash the cathode with dilute nitric acid (1 to 10) adding the washing to the acid solution. Evaporate the contents of the beaker to drive off excess of nitric acid, being careful not to over-heat. Warm the residue with 20 cc. of 20 per cent acetic acid, add 2 cc. of 50 per cent sodium acetate solution, filter and precipitate the lead with 2 cc. of saturated potassium dichromate solution. Allow to stand overnight, filter on a tared Gooch, wash with water, dry for 30 minutes at 125°C., cool and weigh as lead chromate. Multiply the weight of lead chromate by 0.641 to obtain the weight of lead.

35 ARSENIC.—TENTATIVE.

Introduce 5 grams of the sample directly into the generator described under **XI, 2** (Fig. 5), add 10 cc. of water, a little at a time to prevent foaming over, and then 15 cc. of concentrated, arsenic-free hydrochloric acid, introducing it drop by drop until foaming ceases. Heat on a steam bath until a drop of the mixture, when diluted and treated with iodin solution, shows no blue color. Then dilute to about 30 cc. with water, add 4 cc. of potassium iodid solution and continue from this point as directed under **XI, 4**, beginning with "Heat to about 90°C.", except that the blank and the standards for comparison are made by the use of the arsenic-free hydrochloric acid of the same concentration as that used in the determination.

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XXVIII. DRUGS.

CAFFEIN AND ACETANILID IN MIXTURES.

1 PREPARATION OF SAMPLE AND SOLUTION.—TENTATIVE.

(a) If the sample is already in powder form, rub thoroughly in a mortar and keep in a tightly corked tube or flask. Powders in paper, cachet or capsule containers are frequently of such fineness as to require little further trituration except to produce a uniform product. On a tared 5.5 cm. filter weigh 0.3–0.5 gram of the sample or, if preferred, an amount equal to, or a multiple of, the average unit dose (previously ascertained by weighing collectively 20 or more such doses), wash with successive 5–10 cc. portions of the chloroform (30–50 cc. are usually sufficient) until the extraction is complete as indicated by the absence of any residue after evaporation of a small portion of the last washing. Collect the solution in a 200 cc. Erlenmeyer flask, connect the flask with a condenser by means of a cylindrical Kjeldahl connecting bulb[1] and distil until the volume is reduced to about 10 cc.

(b) If the caffein is present in the citrated form, or the composition of the mixture precludes complete extraction as directed in (a), weigh out the desired amount, transfer to a Squibb separatory funnel, add 50 cc. of the chloroform and 20 cc. of water, shake vigorously and, after clearing, draw off the lower layer through a small, dry filter into a 200 cc. Erlenmeyer flask. In the case of coated tablets and pills, ascertain their average weight, powder in a mortar and weigh out for each determination an amount equivalent to one or more tablets or pills. Repeat the extraction twice, using 50 cc. portions of the chloroform for each extraction. Any caffein-acetanilid mixture observable about the apex of delivery tube of the separatory funnel, edge of filter and tip of funnel should be very carefully recovered by judicious washing with chloroform, such washings being subsequently united with the main portion. Distil the combined chloroform extracts to about 10 cc.

(c) In the case of dilute alcoholic solutions, evaporate a measured quantity on a steam bath until most of the alcohol has been expelled, or take an aliquot of the residue from an alcohol determination; transfer to a separatory funnel by pouring and rinsing with a minimum of water so that the final volume does not greatly exceed 20 cc., and then, in order to avoid any loss of acetanilid by hydrolysis during evaporation, add a little solid sodium bicarbonate and a drop of acetic anhydrid. (Should the preparation contain alkaloids, acidify with a few drops of dilute sulphuric acid immediately after acetylization to retain such basic material in the aqueous solution.) Add 50 cc. of the chloroform, shake vigorously, and, after clearing, draw off the chloroform layer through a filter into a 200 cc. Erlenmeyer flask. Repeat the extraction twice, using 50 cc. portions of the chloroform for each extraction, and distil the combined chloroform washings to a volume of about 10 cc.

CAFFEIN AND ACETANILID.—TENTATIVE.

2 REAGENTS.

(a) *Standard bromid-bromate solution.*—Dissolve 50 grams of potassium hydroxid in a small quantity of water, add a slight excess of bromin, dilute with water to dissolve any separated salts, boil to expel excess of bromin and dilute to 1 liter. Standardize the solution against recrystallized and dried acetanilid and adjust the solution so that 1 cc. is equivalent to 5 or 10 mg. of acetanilid as desired.

(b) *Chloroform.*—Redistilled and residue-free. All corks used in the distillation should be treated previously with chloroform.

(c) *Wagner's reagent.*—Dissolve 2 grams of iodin and 6 grams of potassium iodid in a minimum amount of water and dilute to 100 cc.

3

CAFFEIN.—TENTATIVE.

Treat the chloroform extract, obtained in **1**, with 10 cc. of sulphuric acid (1 to 10) and digest on a steam bath until the contents of the flask are reduced to 5 cc. Add 10 cc. of water and continue the digestion until the liquid is again reduced to 5 cc., then cool and transfer to a separatory funnel with a minimum of water, so that the final volume does not greatly exceed 20 cc. Add 50 cc. of the chloroform, extract in the usual way and, after clearing, withdraw the lower layer through a small, dry filter into a 200 cc. Erlenmeyer flask. Repeat the extraction with two 50 cc. portions of the chloroform. Distil the combined extracts down to about 10 cc., finally transferring the residual liquid, by washing with chloroform, to a tared beaker or crystallizing dish. Allow the solution to evaporate spontaneously, or by gentle heat and an air blast, to apparent dryness. Cool and allow to stand in the open until the weight becomes constant.

Chloroform extracts in addition to caffein and acetanilid certain oils, fats, waxes, resins, pigments and other substances from those preparations which contain powdered cinnamon, celery seed, ginger or other vegetable products. These appear either in suspension or solution after the caffein-acetanilid mixture has been digested and contaminate the caffein. Remove any suspended impurities by filtering through a small, moistened filter immediately after hydrolysis and prior to extraction with chloroform. Should the recovered caffein be deeply colored or contaminated with foreign matter, purify it as follows: Dissolve in very dilute sulphuric acid (about 5 cc. of N/5 acid for every 100 mg. of caffein), filter, if necessary, through a moistened filter, add 15–20 cc. of Wagner's reagent, sufficient at least to distinctly color the supernatant liquid a deep claret, stir and allow to stand an hour, preferably in a refrigerator. Filter and wash the periodid with a few cc. of iodin solution, transfer both filter and precipitate to a separatory funnel, using not more than 20 cc. of water, decolorize with a crystal of sodium thiosulphate, then extract with three 50 cc. portions of chloroform and proceed as directed above.

4

ACETANILID.—TENTATIVE.

Transfer the solution of anilin sulphate, remaining in the separatory funnel in **3**, to the Erlenmeyer flask used in effecting hydrolysis, then heat 10 minutes on a steam bath to expel all traces of chloroform. Wash the filter, used in the preceding operation to dry the chloroform solution of caffein, with 5 cc. of water, adding the latter to the main solution of anilin sulphate. Add 10 cc. of concentrated hydrochloric acid, then titrate with the standard bromid-bromate solution until a faint yellow coloration remains, rotating the flask sufficiently to agglomerate the precipitated tribromanilin. Calculate the quantity of acetanilid from the number of cc. required to complete the precipitation.

Caffein and acetanilid are the 2 principal ingredients of the preparation known as "acetanilid compound", a further constituent being sodium bicarbonate. The latter appears as the chloroform-insoluble residue and may be determined by titrating such residue, or one obtained by titrating a portion of the original mixture, with standard acid, using congo red as an indicator. The bicarbonate may also be determined by igniting the original sample, or the chloroform-insoluble residue, with sulphuric acid and weighing the resulting sodium sulphate.

Should the "acetanilid compound" be combined with sodium bromid, the latter, in the absence of other halides, may be determined volumetrically by the Volhard method (II, 17).

CAFFEIN AND ACETPHENETIDIN (PHENACETIN) IN MIXTURES.

5 PREPARATION OF SAMPLE AND SOLUTION.—TENTATIVE.

In the case of preparations containing acetphenetidin instead of acetanilid, but otherwise identical, make the gross separation of the caffein-acetphenetidin mixture as directed under **1**.

6 CAFFEIN.—TENTATIVE.

Treat the chloroform extract, obtained as directed under **1**, with 10 cc. of sulphuric acid (1 to 10) and digest on a steam bath until the liquid is reduced to about 5 cc. Dilute with 10 cc. of water and continue the digestion until the volume is again reduced to 5 cc., again add 10 cc. of water and continue heating until the residual liquid amounts to 8–10 cc. If, during the digestion, particles of acetphenetidin remain on the sides of the flask, rinse them into the solution with a few drops of chloroform.

Great care must also be given to the degree of evaporation. Should the aqueous-acid solution and suspension of caffein-acetphenetidin be concentrated far beyond the limits indicated, more or less phenetidin sulphonate is likely to be formed, which later resists acetylization and conversion to acetphenetidin.

Cool and transfer with water to a separatory funnel, so that the final volume does not greatly exceed 20 cc. Then proceed as directed under **3**.

7 ACETPHENETIDIN.—TENTATIVE.

Wash the filter, used to dry the chloroform in **6**, with 5 cc. of water, receiving the latter in the separatory funnel containing the solution of phenetidin sulphate. Treat with successive small portions of solid sodium bicarbonate until, after complete neutralization of free acid, an excess of the former remains at the bottom of the mixture. Add 50 cc. of chloroform and, for every 0.10 gram of acetphenetidin known or believed to have been present, 5 drops of acetic anhydrid; shake vigorously, allow to clear, then withdraw the chloroform into a second separatory funnel containing 5 cc. of water. Shake this mixture and, after clearing, pass the solvent through a small, dry filter into a 200 cc. Erlenmeyer flask. Distil about 40 cc. of the chloroform, make up the distillate to 50 cc. with chloroform, add this to the material in the first separatory funnel and extract again. Withdraw the chloroform layer to the second separatory funnel wash and distil about 50 cc. (for use in the final extraction). Distil the chloroform down to about 10 cc., transfer with sufficient fresh solvent to a tared 50 cc. beaker or crystallizing dish, evaporate on the steam bath to apparent dryness, finally removing any considerable excess of acetic anhydrid by repeated additions of 1 cc. of chloroform and a drop of alcohol. The reformed acetphenetidin should finally appear as a whitish, crystalline mass with a faint, acetous odor, which disappears completely on standing some hours in the open, or over lime in a vacuum desiccator. Weigh at intervals until the final weight differs from the preceding by not more than 0.5 mg

CAFFEIN AND ANTIPYRIN IN MIXTURES[1].

8 PREPARATION OF SAMPLE AND SOLUTION.—TENTATIVE.

(**a**) Extract a weighed portion of the finely powdered sample on a filter with chloroform to separate the caffein and antipyrin from the usual excipients of tablet and pill combinations. Distil the greater part of the chloroform and evaporate the remainder on the steam bath.

(**b**) In the case of alcoholic preparations, remove the alcohol from a measured amount of the sample by heating on a steam bath. Extract the residue with three 50 cc. portions of chloroform in a separatory funnel. Distil the greater portion of the chloroform and evaporate the remainder on a steam bath.

9 ANTIPYRIN.—TENTATIVE.

Transfer the residue, obtained in 8, which should weigh about 0.25 gram, to a 150 cc. separatory funnel by means of two 5 cc. portions of alcohol-free chloroform, followed by 10 cc. of water. Add 1 gram of sodium bicarbonate and 10–15 cc. of N/5 iodin (or double the quantity of N/10 iodin), adding the latter in small portions and shaking the mixture vigorously after each addition. The iodin should then be in excess of that required to convert all the antipyrin into the mono-iod derivative. If not, add a little more and shake the mixture again. Remove the free iodin with a small crystal of sodium thiosulphate and add 15 cc. of chloroform, shaking vigorously for 1 minute. After clearing, draw off the chloroform solution into a second separatory funnel, wash with 5 cc. of water, filter through a small, dry filter into a tared 50 cc. beaker and evaporate to apparent dryness on the steam bath, using an air blast. Repeat the extraction with two (three, if N/10 iodin is used) 25 cc. portions of chloroform, wash, filter and evaporate each portion as above. Recover any crystalline product separating about the tip of the delivery tube and funnel and edge of filter by judicious washing with chloroform. Dry the nearly colorless, crystalline residue of caffein and iodantipyrin 30 minutes at 105°C., cool and weigh. Designate this weight as "a".

The use of alcohol-free chloroform in connection with the halogenation of antipyrin is necessary in order to preclude the formation of iodoform, the presence of which in the composite residue (a) would vitiate the result.

Dissolve the composite residue in 5 cc. of glacial acetic acid, add 10 cc. of saturated sulphur dioxid solution, then transfer with hot water to a 400–500 cc. beaker until the final volume amounts to about 200 cc. Add sufficient silver nitrate solution to precipitate all the iodin (about 0.3 gram of silver nitrate); then a few drops of nitric acid, heat nearly to boiling and stir to agglomerate the silver iodid. Add 15 cc. of concentrated nitric acid, cover the beaker with a watch glass and boil gently for 5 minutes. Filter by decantation through a tared Gooch, wash the precipitate once with a little alcohol, then with two 100 cc. portions of boiling water and finally transfer the iodid to the crucible. Wash several times with hot water and again with alcohol to remove traces of organic matter, dry 30 minutes in an air bath at 110°C., cool and weigh. The weight of silver iodid multiplied by 0.8012 gives the weight of antipyrin.

10 CAFFEIN.—TENTATIVE.

Calculate the quantity of caffein by multiplying the weight of silver iodid by 1.3374 and subtracting the product from the weight "a" above.

In the analysis of a mixture containing caffein, antipyrin, acetanilid and sodium salicylate, the following steps are essential in effecting a separation: (1) Extraction of caffein, acetanilid and antipyrin with chloroform from the aqueous soda solution; (2) Hydrolytic treatment with sulphuric acid of the 3 substances thus separated preliminary to the determination of caffein and antipyrin as directed under 9 and 10.

ACETANILID AND ACETPHENETIDIN (PHENACETIN) IN MIXTURES[1].

ACETPHENETIDIN.—TENTATIVE.

11 REAGENTS.

(a) *Purified iodin.*—Dissolve 2 parts of resublimed iodin and 1 of potassium iodid in 1 of water, pour the clear solution into a large volume of water, filter and wash the finely precipitated iodin several times on a porous plate with water. Dry in the air and finally in a desiccator containing sulphuric acid where it is kept in a glass-stoppered weighing bottle.

(b) *Standard sodium thiosulphate solution.*—Dissolve 30 grams of crystallized sodium thiosulphate in water and dilute to 1 liter. Standardize this solution against the purified iodin as follows: Weigh out about 0.3 gram of the purified iodin in a small, glass capsule (about ½ inch high and ⅜ inch in diameter), provided with a closely fitting glass cap or stopper, and place the capsule in a 200 cc. Erlenmeyer flask containing 0.5 gram of potassium iodid dissolved in 1–2 cc. of water. After complete solution, dilute with 10 cc. of water and titrate with the sodium thiosulphate solution, using 1 or 2 drops of starch solution as an indicator.

(C) *Standard iodin solution.*—Dissolve 40 grams of potassium iodid in the least possible quantity of water, add 30 grams of the purified iodin and, after solution, dilute to 1 liter. Standardize 25 cc. of this solution against the standard sodium thiosulphate solution.

12 DETERMINATION.

(1) Place 0.2 gram of the acetphenetidin-acetanilid mixture in a 50 cc. lipped Erlenmeyer flask, add 2 cc. of glacial acetic acid, heat gently over a wire gauze to complete solution and dilute with 40 cc. of water, previously warmed to 70°C. Transfer the clear liquid with two 10 cc. portions of warm (40°C.) water to a glass-stoppered, 100 cc. graduated flask containing 25 cc. of the standard iodin solution warmed to 40°C. Stopper, mix thoroughly by rotating the liquid, then add 3 cc. of concentrated hydrochloric acid, continue rotating the liquid until crystallization begins and then set aside to cool. If the ratio of acetphenetidin to acetanilid is equal to or greater than unity, crystalline scales will form almost immediately on the addition of acid. As the proportion of acetanilid increases, however, the periodid tends to remain in the liquid state. In such cases, gentle agitation or rotation of the flask in water, warmed not to exceed 40°C., hastens the formation of crystals. When the contents of the flask are at room temperature, fill with water to within 2–3 cc. of the mark, mix thoroughly by rotating the mixture and allow to stand overnight. Fill to the mark with water, mix thoroughly, allow to stand 30 minutes, filter through a 5.5 cm. dry, closely fitted filter into a 50 cc. graduated flask, rejecting, however, about 15 cc. of the first runnings but reserving them for the recovery of acetanilid. Transfer the 50 cc. aliquot to a 200 cc. Erlenmeyer flask and titrate with the standard sodium thiosulphate solution. Calculate the amount of acetphenetidin from the following formula:

Acetphenetidin = I (0.0896 × N) in which

0.0896 = the quantity of acetphenetidin contained in 1 cc. of a normal solution of this substance;

N = the normality of the standard sodium thiosulphate solution employed; and

I = the number of cc. of the standard sodium thiosulphate solution corresponding to the iodin combined with the acetphenetidin.

The formula of the precipitated periodid, which constitutes the basis for the above determination, is $(C_2H_5O.C_6H_4NH.COCH_3)_2HI.I_4$.

(2) The gravimetric determination of acetphenetidin may, if desired, be effected as follows: Filter off the periodid, preferably by suction, wash with 10–15 cc. of the standard iodin solution, then transfer the precipitate together with the filter, likewise any particles of the precipitate remaining in the graduated flask, to a separatory funnel, using not over 50 cc. of water. Remove both free and added iodin with a few small crystals of sodium sulphite and extract the liquid with three 50 cc. portions of chloroform, washing each portion subsequently into a second separatory funnel with 5 cc. of water. After washing and clearing, filter the chloroform solution through a dry 5.5 cm. filter into a 200 cc. Erlenmeyer flask, distil most of the chloroform, transfer the residual solution (5–10 cc.), by means of a little chloroform, to a small, tared beaker or crystallizing dish, evaporate to dryness on a steam bath, cool and weigh.

For the identification of acetphenetidin, either alone or in admixture with acetanilid, the following test will be found of value[3]: To 1–2 mg. of the sample in a test tube add a drop of acetic acid, 0.5 cc. of water and 1 cc. of N/10 iodin, warm the mixture to about 40°C., then add a drop of concentrated hydrochloric acid. If acetphenetidin alone is present, its periodid separates almost immediately in the form of reddish brown leaflets or needle-like crystals. If the sample consists largely of acetanilid, the separation takes place on cooling and shaking the liquid. In the presence of considerable acetanilid, the periodid first separates as minute, oily globules, which, on vigorous shaking, gradually become crystalline. This test is so delicate that as little as 0.5 mg. of acetphenetidin may, if alone, be detected in the form of its characteristic periodid.

13 ACETANILID.—TENTATIVE.

(1) If the combined weight of the acetphenetidin-acetanilid mixture is known, determine that of the latter ingredient by difference; or (2), determine it directly from a second aliquot of the filtrate from the acetphenetidin periodid in **12** as follows:

Pipette 25–30 cc. of the clear liquid into a separatory funnel, decolorize with solid sodium sulphite and solid sodium bicarbonate in slight excess, add 1 or 2 drops of acetic anhydrid, then extract with three 60 cc. portions of chloroform, passing the chloroform solution, when cleared, through a small, dry filter into a 200 cc. Erlenmeyer flask, and distil the chloroform, by the aid of gentle heat, to about 20 cc. Add 10 cc. of sulphuric acid (1 to 10) and digest on a steam bath until the residue has been reduced one-half, add 20 cc. of water and continue the digestion for an hour; then add a second 20 cc. portion of water and 10 cc. of concentrated hydrochloric acid, titrate very slowly, drop by drop, with the standard bromid-bromate solution, **2** (**a**), until a faint yellow color remains. While adding this reagent, rotate the flask sufficiently to agglomerate the precipitated tribromanilin. Calculate the amount of acetanilid present.

If the preparation contains caffein or antipyrin or both in addition to acetanilid and acephenetidin, proceed as follows: (1) Digest the mixture by heating with dilute sulphuric acid to convert acetphenetidin and acetanilid to phenetidin and anilin sulphates, respectively; (2) Separate the caffein and antipyrin by extraction with chloroform; (3) Re-form acetphenetidin and acetanilid by treating the solution of the corresponding sulphates with solid sodium bicarbonate in slight excess, then add a few drops of acetic anhydrid and extract with chloroform[4].

ACETPHENETIDIN (PHENACETIN) AND SALOL IN MIXTURES[5].

ACETPHENETIDIN.

14 *Acid Hydrolysis Method.—Tentative.*

Weigh out on a tared 5.5 cm. filter an amount of the sample equal to, or a multiple of, the average weight of a unit dose and wash with sufficient successive, small portions of chloroform to extract completely all acetphenetidin and salol present in the mixture (about 40 cc.). Collect the solution in a tared, 100 cc. beaker and evaporate on a warm plate (50°–60°C.) to apparent dryness, using an air blast. Let stand 24 hours at room temperature to practically constant weight, then transfer the crystalline residue, by means of chloroform, to a 50 cc. lipped Erlenmeyer flask, evaporate the solvent by means of an air blast and gentle heat, add 10 cc. of sulphuric acid (1 to 10) and evaporate on the steam bath until the volume is reduced one-half. Add 10 cc. of water and continue the digestion as before, then add a second 10 cc. of water and evaporate to 5 cc. Transfer the residue with about 20 cc. of water to a small separatory funnel and extract with 15, 10 and 5 cc. of chloroform, washing each extract with 5 cc. of water in a second separatory funnel to recover traces of phenetidin sulphate possibly dissolved by the chloroform, finally rejecting the latter since it contains all the salol not previously eliminated during the digestion.

Add the wash water in the second separatory funnel to the solution of phenetidin sulphate in the first separatory funnel and proceed as directed under **7**, beginning with "Treat with successive small portions of solid sodium bicarbonate".

15 *Alkaline Hydrolysis Method.—Tentative.*

On a small, tared filter weigh out an amount of the sample to contain not more than 0.10 gram of salol, exhaust with chloroform as directed in **14**, collect the solvent in a small, lipped Erlenmeyer flask and evaporate the chloroform by means of an air blast without heat. Add 10 cc. of 2.5 per cent sodium hydroxid solution and heat 5 minutes on a steam bath. Cool quickly to room temperature in running water to prevent partial hydrolysis of the acetphenetidin. Transfer the liquid to a separatory funnel with a minimum of water, then rinse out the flask with the first 20 cc. portion of chloroform used in the extraction. Extract the alkaline solution with three 20 cc. portions of chloroform, wash each portion in a second separatory funnel with 5 cc. of water and pass the solution through a small, dry filter into a 200 cc. Erlenmeyer flask. Designate the combined alkaline solution and washings as *A*. Distil the combined chloroform extracts to about 5 cc. Transfer by means of a little chloroform to a small, tared beaker or crystallizing dish, evaporate on a steam bath with the aid of an air blast, cool and weigh the residual acetphenetidin at intervals until the weight becomes constant.

SALOL.

16 *Acid Hydrolysis Method.—Tentative.*

Subtract the weight of acetphenetidin, as determined in **14**, from the combined weight of the 2 ingredients determined in **14**, to obtain the weight of salol.

17 *Alkaline Hydrolysis Method.—Tentative.*

Place the combined alkaline solutions *A*, under **15**, in a 500 cc. glass-stoppered bottle, dilute with water to about 200 cc., run in from a burette an excess (about 45 cc.) of N/7 potassium bromid-bromate, add 10 cc. of concentrated hydrochloric acid and shake 1 minute, then at intervals for 30 minutes. Add 10 cc. of 15 per cent potassium iodid solution and shake at intervals for 15 minutes. Titrate the free iodin with standard sodium thiosulphate solution (preferably N/7), previously standardized against the N/7 bromid-bromate solution. One cc. of N/7 potassium bromid-bromate is equivalent to 2.55 mg. of salol. From the number of cc. of the N/7 bromid-bromate solution used, calculate the amount of salol on the basis of 12 atoms of bromin to 1 molecule of salol.

ACETANILID AND SODIUM SALICYLATE IN MIXTURES.

18 PREPARATION OF SAMPLE AND SOLUTION.—TENTATIVE.

Weigh an amount of the powdered sample equal to, or a multiple of, an average unit dose, transfer to a separatory funnel containing 10 cc. of water, and, for every unit dose, add 0.10 gram of solid sodium bicarbonate. In the case of coated tablets and pills, ascertain their average weight, powder in a mortar and weigh out an amount of the powder equivalent to one or more tablets or pills for each determination prior to treatment in the separatory funnel. In the examination of alcoholic preparations, distil the alcohol from a measured volume on a steam bath, transfer to a separatory funnel with a minimum of water and add sufficient solid sodium bicarbonate (0.5 to 1.0 gram).

19 ACETANILID.—TENTATIVE.

Extract the alkaline solution, obtained under **18**, with three 50 cc. portions of chloroform, wash each portion with 5 cc. of water in a second separatory funnel and collect the solvent, without previous drying, in a 200 cc. Erlenmeyer flask. Designate the aqueous solution as *A*. Distil the chloroform very gently to about 5 cc., add 10 cc. of dilute sulphuric acid and completely hydrolyze on the steam bath. Proceed from this point as directed under **4**.

20 SODIUM SALICYLATE.—TENTATIVE.

Acidify the aqueous solution of sodium salicylate *A*, under **19**, with a few drops of concentrated hydrochloric acid and extract with sufficient (3–5) 25 cc. portions of chloroform to exhaust the salicylic acid present in the mixture. Treat each portion in a second separatory funnel with 20 cc. of water, containing 1 gram of anhydrous sodium carbonate for every 100 mg. of salicylic acid. Shake vigorously and, after clearing, wash each portion again in a second separatory funnel with 5 cc. of water, then add the washings to the main aqueous soda solution of sodium salicylate. Dilute to a known volume, transfer an aliquot, representing about 100 mg. of salicylic acid, to a 200 cc. Erlenmeyer flask, make up to 100 cc., heat nearly to boiling, then add slowly 25–40 cc. of strong (about N/5) iodin solution, sufficient to insure an excess during digestion, and digest for an hour on a steam bath. Remove the free iodin with a few drops of sodium thiosulphate solution, decant the clear liquid through a tared Gooch, retaining most of the precipitate, tetraiodophenylenequinon, $(C_6H_2I_2O)_2$, in the flask. To the latter add 50 cc. of boiling water, digest 10 minutes on the steam bath, then filter, wash gradually all the precipitate into a Gooch, using for this purpose and the final washing about 200 cc. of hot water. Dry to constant weight in an air bath at 100°C. Multiply the weight of the precipitate by 0.4654 to obtain the quantity of sodium salicylate present in the aliquot taken.

Should the mixture contain caffein or antipyrin, or both, these substances will appear with the acetanilid in the first chloroform extract and may be determined as directed in closely set type following **10**. Should the acetanilid be replaced by acetphenetidin in the mixture, the general procedure would not be materially altered, the acetphenetidin being weighed directly after recovery from its washed chloroform solution as separated from the sodium salicylate. If, instead of sodium salicylate, the mixture contains the free acid or its ammonium salt, add a larger quantity of sodium bicarbonate prior to extraction with chloroform to insure the fixation of salicylic acid.

In the analysis of a mixture of caffein, acetanilid, sodium salicylate and codein sulphate, the following procedure is recommended: (1) Extraction of caffein, acetanilid and salicylic acid from the acidified solution; (2) Washing the chloroform solution with aqueous soda solution for the recovery of salicylic acid, preliminary to its treatment with iodin solution; (3) Separation of caffein and acetanilid as directed under 3 and 4; (4) Recovery of codein from the solution of its sulphate after treatment with sodium bicarbonate and chloroform.

CAFFEIN, ACETANILID AND QUININ SULPHATE IN MIXTURES.

21 PREPARATION OF SAMPLE AND SOLUTION.—TENTATIVE.

Transfer to a separatory funnel one or more average unit doses of the powdered sample, add 20 cc. of water and 50 cc. of chloroform, then 10 drops of dilute sulphuric acid and extract in the usual way. After clearing, wash the solvent in a second separatory funnel with 5 cc. of water prior to transferring to a 200 cc. Erlenmeyer flask. Repeat the foregoing operations with two 50 cc. portions of chloroform, finally distilling the combined chloroform solution by gentle heat to about 10 cc.

22 CAFFEIN AND ACETANILID.—TENTATIVE.

Treat the chloroform residue obtained in **21** as directed under **3** and **4**.

23 QUININ SULPHATE.—TENTATIVE.

Combine the wash water, used in the second separatory funnel in **21**, with the solution of quinin bisulphate, add a slight excess of solid sodium bicarbonate, extract with three 50 cc. portions of chloroform, wash each portion with 5 cc. of water in a second separatory funnel and then pass through a dry filter into a 200 cc. Erlenmeyer flask. Distil by gentle heat to about 5 cc., evaporate on a steam bath to apparent dryness, dissolve the amorphous alkaloid in 5 cc. of neutral alcohol and titrate with N/50 hydrochloric acid to a faint red, using 2 drops of methyl red as an indicator. Heat on a steam bath until most of the alcohol has been expelled, adding, if necessary, sufficient acid to maintain the acid reaction. From the total number of cc. of acid employed in the titration calculate the quinin sulphate. One cc. of N/50 hydrochloric acid is equivalent to 8.73 mg. of quinin sulphate.

If the mixture contains acetphenetidin in place of acetanilid, proceed as outlined above, except that the separation of caffein and acetphenetidin is conducted as directed under 6 and 7.

CAFFEIN, ACETANILID AND CODEIN SULPHATE IN MIXTURES.

24 PREPARATION OF SAMPLE AND SOLUTION.—TENTATIVE.

Proceed as directed under **21**.

25 CAFFEIN AND ACETANILID.—TENTATIVE.

Proceed as directed under **22**.

26 CODEIN SULPHATE.—TENTATIVE.

Proceed as directed under **23** to the point indicated by the sentence "Distil by gentle heat to about 5 cc.". Transfer the chloroform solution of codein with sufficient solvent to a small, tared beaker, evaporate to apparent dryness on a steam bath, add a few drops of alcohol to the amorphous residue, then a like amount of water and evaporate again. Finally cool and allow the usually crystalline product to stand until the weight becomes constant. The weight of this residue multiplied by 1.314 gives the quantity of codein sulphate present.

This result should be checked volumetrically. Dissolve the residue in 3–5 cc. of neutral alcohol and titrate with N/50 sulphuric acid to a faint red, using methyl red as an indicator. From the number of cc. of standard acid employed calculate the amount of codein sulphate. One cc. of N/50 sulphuric acid is equivalent to 7.87 mg. of codein sulphate. The quantity of codein sulphate as found by weight will usually be slightly greater than that determined by titration.

CAFFEIN, ACETANILID, QUININ SULPHATE AND MORPHIN SULPHATE IN MIXTURES.

27 PREPARATION OF SAMPLE AND SOLUTION.—TENTATIVE.

Transfer to a separatory funnel an amount (containing not less than one-fourth grain of morphin) of the powdered sample equal to, or a multiple of, a unit dose, add 20 cc. of water and 10 drops of dilute sulphuric acid, then extract with three 50 cc. portions of alcohol-free chloroform, wash each portion in a second separatory funnel with 5 cc. of water and add the combined washings to the alkaloidal solution in the first separatory funnel. Filter the chloroform extracts through a small, dry filter into a 200 cc. Erlenmeyer flask and distil by gentle heat to about 10 cc.

28 CAFFEIN AND ACETANILID.—TENTATIVE.

Treat the chloroform residue as directed under **3** and **4**.

29 QUININ SULPHATE.—TENTATIVE.

Add to the solution of quinin and morphin sulphates, obtained in **27**, 4–5 cc. of sodium hydroxid solution (1 to 10) and extract with four 40 cc. portions of chloroform, wash each portion with 5 cc. of water and pass the clear solvent through a small, dry filter into a 200 cc. Erlenmeyer flask. Remove the solvent by gentle distillation and titrate the residual quinin with N/50 hydrochloric acid as directed under **23**.

If the morphin sulphate present is contaminated with codein sulphate, the latter will be separated and weighed with the quinin.

30 MORPHIN SULPHATE.—TENTATIVE.

Wash the filter, employed in **29**, with 5 cc. of water and add to the aqueous alkaline solution of the alkaloid. Now add 0.5 gram of ammonium chlorid (or an amount slightly in excess of that required to free the morphin as well as convert all sodium hydroxid to sodium chlorid) and, to the resulting ammoniacal solution, add 45 cc. of chloroform and 5 cc. of alcohol, then extract in the usual way, washing the solvent in a second separatory funnel with 5 cc. of water. After clearing, pass the chloroform through a small, dry filter into a 200 cc. Erlenmeyer flask. Repeat the extraction with three 40 cc. portions of chloroform, washing and filtering as before, finally collecting all the solvent in an Erlenmeyer flask and distilling to about 10 cc. Transfer with chloroform to a small, tared beaker, evaporate to apparent dryness, add 0.5 cc. each of water and neutral alcohol, start crystallization by stirring with a glass rod and finally evaporate to dryness. Cool and allow to stand until the weight becomes constant.

Check the weight of morphin, thus determined, by titration with N/50 sulphuric acid, using a drop of methyl red as an indicator. Dissolve the alkaloid in 1–2 cc. of warm, neutral alcohol, then add the standard acid to a faint red. Evaporate most of the alcohol on a steam bath, adding, if necessary, sufficient acid to maintain the acid reaction. From the volume of acid used calculate the morphin sulphate. One cc. of N/50 sulphuric acid is equivalent to 7.58 mg. of morphin sulphate.

Despite all precautions looking to the exclusion of impurities from the morphin as weighed, the amount of this substance thus determined will usually be greater than that found volumetrically. In order to insure the greatest possible accuracy in volumetric operations on alkaloidal residues like quinin, morphin, and codein, it is suggested that whenever possible the strength of the standard acid used be checked by titration against the pure alkaloid under examination.

In the various operations involving fixation and subsequent liberation of morphin by means of fixed alkali and ammonium chlorid, the most careful attention should be paid to the manner of adding the reagents, since any undue excess of either might nullify the entire procedure. Any large excess of sodium hydroxid would naturally require for its reduction a correspondingly large amount of ammonium chlorid, the latter in turn yielding its equivalent of hydroxid, relatively large quantities of which through interaction with sodium chlorid tend to inhibit any permanent liberation of alkaloid and thus prevent complete extraction. Furthermore, ammonium chlorid in large amount operates retentively on the morphin in solution, due in part possibly to the formation of an alkaloidal hydrochlorid.

31 STRYCHNIN IN LIQUIDS.

(Applicable to elixirs of iron and strychnin, in absence of other alkaloids.)

Measure 50 cc. of the sample into an evaporating dish and remove alcohol by evaporation. Transfer to a 250 cc. Squibb separatory funnel and add a slight excess of ammonium hydroxid. Then add 25 cc. of chloroform, agitate and allow to stand until separation is complete. Draw off the chloroform solution into a second separatory funnel and repeat the extraction with two 25 cc. portions of the solvent. Wash the combined chloroformic fractions with 10 cc. of water and allow to stand 15 minutes. Introduce a pledget of absorbent cotton into the stem of the separatory funnel and carefully draw off the chloroform solution into a tared dish, but do not allow the wash water to enter the orifice of the stop-cock. Add 10 cc. of chloroform to the contents of the separatory funnel and agitate. Allow to stand until separation is complete, then draw off the chloroform into the tared dish and wash the outer surface of the stem of the separatory funnel with a few cc. of chloroform. Evaporate on a steam bath, removing the dish from the bath as the last portions evaporate to avoid decrepitation. Dry at 100°C. to constant weight and weigh as strychnin.

32 STRYCHNIN IN TABLETS.

Weigh 25 tablets and introduce directly into a 250 cc. Squibb separatory funnel. Moisten with 8 cc. of water and then add 1 cc. of ammonium hydroxid. From this point proceed as directed under **31**, beginning with "Then add 25 cc. of chloroform, agitate and allow to stand until separation is complete".

33 ATROPINE IN TABLETS.

Weigh 25 tablets and introduce directly into a small separatory funnel. Moisten with 5 cc. of water. Add 1 cc. of ammonium hydroxid. Agitate with 25 cc. of chloroform and allow to stand until separation is complete. Draw off the chloroform layer into a second separatory funnel and repeat the agitation with two 25 cc. portions of the solvent. After combining all of the fractions, wash the combined chloroformic solutions by agitation with 10 cc. of water and allow to stand 15 minutes. Introduce a pledget of absorbent cotton into the stem of the separatory funnel and carefully draw off the chloroform solution into a tared dish, but do not allow the wash water to enter the orifice of the stop-cock. Add 10 cc. of chloroform and when the water has entirely risen to the surface, draw off the chloroform into the tared dish. Wash the outer surface of the stem of the separatory funnel with a little chloroform. Evaporate on a steam water-bath, removing the dish from the bath as the last portions evaporate to avoid decrepitation. Dry in vacuo to constant weight and weigh as atropine.

Check the weight of atropine by dissolving the residue in neutral alcohol, adding an excess of N/10 sulphuric acid and titrating back with N/50 potassium hydroxid, using methyl red as an indicator. One cc. N/50 $H_2SO_4 = .005784$ gm. atropine.

TRAGACANTH.

34 VOLATILE ACIDITY[6].—TENTATIVE.

The quantity of volatile (acetic) acidity developed in the acid hydrolysis of gum tragacanth (*Astragalus gummifer*) affords a valuable index of the purity of this commodity when compared with results obtained by similar treatment of so-called "Indian gum" (*Cochlospernum gossypium and Sterculia urens*). The term "volatile acidity" expresses the number of cc. of N/10 potassium or sodium hydroxid required to neutralize the volatile (acetic) acid obtained by distilling with steam the products of the action of boiling aqueous phosphoric acid on 1 gram of the gum.

Treat 1 gram of the whole or powdered sample in a 700 cc. round-bottomed, long-necked flask for several hours in the cold with 100 cc. of water and 5 cc. of sirupy phosphoric acid until the gum is completely swollen. Boil gently for 2 hours under a reflux condenser. A very small amount of cellulose substance will remain undissolved. Now distil the hydrolyzed product with steam, using a spray trap[7] to connect the distillation flask with the condenser and continue until the distillate amounts to 600 cc. and the acid residue to about 20 cc. Do not concentrate too far, as this would scorch the non-volatile, organic decomposition products and possibly contaminate the distillate. Titrate the distillate with N/10 potassium hydroxid, using 10 drops of phenolphthalein as an indicator, finally boiling the liquid under examination until a faint pink color remains. Correct the result by a blank determination and express the final result in terms of the number of cc. of N/10 alkali required, as in the above definition.

While tragacanth yields a practically colorless solution when boiled with aqueous phosphoric acid, Indian gum, on the other hand, gives a pink or rose solution. This reaction may be used as a preliminary test for the detection of Indian gum.

LEVANT WORMSEED.

35 SANTONIN.—TENTATIVE.

Extract 10 grams of the sample, ground to pass a 30 mesh sieve, in a Soxhlet extraction apparatus for 3 hours with chloroform. Distil the chloroform until 7–8 cc. remain; add 100 cc. of 5 per cent barium hydroxid solution and heat on a steam bath until the odor of chloroform has disappeared. Boil 5 minutes, cool and pass carbon dioxid (washed through sodium bicarbonate solution to remove traces of acid) until saturated. Filter on a small Büchner funnel, using suction, and wash twice with 10 cc. of water. Heat the filtrate on a steam bath, add 5 cc. of 25 per cent hydrochloric acid (sp. gr. 1.125) and warm 5 minutes. Cool until lukewarm and extract with 20, 15 and 15 cc. of chloroform, passing the solvent through a small filter into a flask. Evaporate to dryness, removing the last traces of chloroform. Dissolve in 7.5 grams (9.5 cc.) of absolute alcohol, warming gently if necessary. Then add 42.5 cc. of water heated to 60°-70°C., stopper the flask and allow to cool. Start crystallization at this point by scratching the side of the flask with a rod or by seeding with a minute crystal of santonin. (Solutions containing a liberal amount of santonin, kept in a cool place for 24 hours, have been found in a supersaturated condition where this precaution was not observed.) Maintain the flask and contents at a temperature of 15°–17°C. for 24 hours. Filter and wash at 15°–17°C. with two 10 cc. portions of 15 per cent alcohol by weight. Dry the flask and filter at 100°C., dissolve the santonin left in the flask and on the filter in chloroform and filter into a tared beaker. Wash the flask and paper thoroughly with chloroform, evaporate the combined filtrate and washings, dry at 100°C. to remove all traces of chloroform and weigh. To the weight found add 0.04 gram for the santonin dissolved in the dilute alcohol and multiply the total by 10 to obtain the per cent of santonin.

NITROGLYCERIN IN TABLETS.

36 PREPARATION OF SAMPLE.—TENTATIVE.

(a) Crush 25 tablets under 10 cc. of anhydrous ether in a 25 cc. cylinder by means of a stout glass rod. Rinse the rod with a little anhydrous ether, allow the insoluble material to settle and decant the solution into a 50 cc. graduated flask. Wash the residue repeatedly with 5 cc. portions of anhydrous ether, decant the washings into the flask until it is filled to the mark, stopper and mix well. Designate this solution as *A*.

Add 10 cc. of water to the residue, mix well and transfer the mixture to a small separatory funnel by means of a little water. Extract with 3 successive portions of 10, 5 and 5 cc. of ether. Collect the ether extracts in a 50 cc. beaker and designate this solution as *B*.

(**b**) Disintegrate 25 tablets in a small beaker with 10 cc. of water, breaking up any lumps with a glass rod, and transfer by means of a little water to a separatory funnel. Rinse the beaker with 10 cc. of ether and transfer this also to the separatory funnel. Shake thoroughly, draw off the aqueous layer and transfer the ether through a funnel, containing a little cotton, to a 50 cc. graduated flask. Repeat the extraction with successive portions of ether until the flask is filled to the mark, stopper and mix well. Designate this solution as *C*.

In hand-made and soft compressed tablets, the method described under (a) is preferred, since the direct extraction of the dry crushed material with ether removes most of the nitroglycerin. In hard compressed tablets, the direct extraction is often not nearly so complete and, in such cases, the method described under (b) is to be preferred.

Nitrate Method[8].—*Tentative*.

37 REAGENTS.

(**a**) *Phenoldisulphonic acid solution*.—Prepare as directed under **III**, **14** (**a**).

(**b**) *Standard nitrate solution*.—Dissolve 0.7217 gram of potassium nitrate in 1 liter of water. Evaporate 10 cc. of this solution just to dryness in a porcelain dish on a steam bath. Cool the residue and treat it with 2 cc. of the phenoldisulphonic acid solution, rubbing with a glass rod to insure intimate contact. After 5–10 minutes dilute to 250 cc. Each cc. of this solution contains 0.004 mg. of nitrogen. Add an excess of potassium hydroxid solution to an aliquot of this solution and dilute to 100 cc. (Do not use sodium or ammonium hydroxid.) It is advisable to prepare a standard of approximately the same color as the unknown.

38 DETERMINATION.

Place 20 cc. of the ether solution, *A* or *C*, obtained in **36**, in a dried, tared 50 cc. beaker. Evaporate the solvent in a vacuum desiccator containing sulphuric acid. Apply the vacuum gradually, to prevent boiling. Allow the beaker to remain in the vacuum 30 minutes after the ether has evaporated. Weigh and calculate the ether extract per tablet. Treat the residue with 2 cc. of the phenoldisulphonic acid solution, rotating the beaker so that the reagent comes in contact with the entire inner surface. After 10 minutes add water and wash into a 100 cc. flask. Dilute to the mark and place 10 cc., representing 1 tablet, in a 100 cc. flask, add about 50 cc. of water and a few drops more of 20 per cent potassium hydroxid solution than is required to neutralize the acid. Dilute to the mark and compare the color with that produced when a standard nitrate solution is similarly treated. Any convenient colorimeter or Nessler tubes may be used. Multiply the nitrate nitrogen found by 5.4 to obtain the equivalent of nitroglycerin.

When the sample is prepared as directed under **36** (**a**), a correction (determined as directed in **40**), should be made for the amount of nitroglycerin in *B* under **36**, using all of *B* instead of an aliquot.

Nitrite Method[9].—*Tentative*.

39 REAGENTS.

(**a**) *Sulphanilic acid solution*.—Prepare as directed under **III**, **12** (**b**).

(**b**) *Alpha-naphthylamin hydrochlorid solution*.—Prepare as directed under **III**, **12** (**c**).

(c) *Standard nitrite solution.*—Weigh out 0.220 gram of dry silver nitrite, XIV, 19 (c), dissolve in a small quantity of hot water and decompose with a slight excess of sodium chlorid solution. When the solution becomes clear, dilute to 1 liter with nitrite-free water. Dilute 5 cc. of this solution to 1 liter with nitrite-free water. The second dilution, containing 0.0001 mg. of nitrous nitrogen per cc., is the standard to be used. [Cf. III, 12 (d).]

40

DETERMINATION.

Place 5 cc. of the ether solution, *A* or *C*, under 36, in a 50 cc. beaker, dilute with 5–10 cc. of alcohol and add about 5 cc. of 0.5 per cent alcoholic potassium hydroxid. Cover with a watch glass and allow to stand 10 minutes. Place on a steam bath, boil, remove the watch glass and, when most of the liquid has evaporated, add about 25 cc. of water and return to the steam bath until about half the liquid has evaporated or until the odor of alcohol can no longer be detected. Cool and dilute with nitrite-free water to 250 cc. Each cc. of this solution represents 0.01 of a tablet. Introduce an aliquot, representing 0.02–0.04 mg. of nitroglycerin, into a 100 cc. graduated flask, dilute with sufficient nitrite-free water to make the volume 90–95 cc., add a drop of concentrated hydrochloric acid, then 2 cc. of the sulphanilic acid solution and 2 cc. of the alpha-naphthylamin hydrochlorid solution. Complete the volume with nitrite-free water. Prepare at the same time and in the same manner standards containing known amounts of sodium nitrite. Stopper the flasks, mix well and compare the colors after 30 minutes, using any convenient colorimeter or Nessler tubes. Multiply the nitrite nitrogen found by the factor 8, which has been determined experimentally, to obtain the equivalent of nitroglycerin.

When the sample is prepared as directed under 36 (a), a correction, determined as directed above, should be made for the amount of nitroglycerin in *B* under 36, using all of *B* instead of an aliquot.

PEPSIN IN LIQUIDS.—TENTATIVE.

41

REAGENTS.

(a) *Standard pepsin.*—Powder a good grade of U. S. P. pepsin and pass it through a No. 60 sieve; dry in vacuo over calcium chlorid, again pass through a sieve and preserve in a stoppered bottle. Ascertain the exact pepsin equivalent of the dry powder by the U. S. P. method[10] and express in percentage based on the assumption that the U. S. P. product is 100 per cent pure.

(b) *Standard pepsin solutions.*—Weigh out definite amounts of the standard pepsin into the requisite quantity of N/10 hydrochloric acid to make solutions containing 5 and 0.5 mg. of pepsin per cc. These should be freshly prepared.

(c) *Ricin solution.*—Grind commercial ricin, similar to the "Ricin Prepärat nach Jacoby", to a No. 60 powder, mix thoroughly, dry and keep in a desiccator. Digest 1 gram of this powder for an hour at 37.5°C. in 100 cc. of 5 per cent sodium chlorid solution, cool, filter and use at once for the assay.

42

PREPARATION OF SOLUTIONS.

(a) *Dilute solution of the sample.*—Dilute the sample with a measured amount of N/10 hydrochloric acid until, upon digestion at 37.5°C., 1 cc. requires approximately 15 minutes to digest the precipitate obtained by mixing 2 cc. of the ricin solution and 0.5 cc. of N/10 hydrochloric acid. To 50 cc. of this diluted preparation add the requisite quantity of water or of N/5 hydrochloric acid to make the preparation of N/10 acid strength when diluted with N/10 acid to 90 cc. Preserve the sample in a refrigerator. (Solid pepsin preparations may often be extracted with hydrochloric acid of appropriate strength and prepared for assay in the same manner.)

(b) *Dilute comparison solution of the sample.*—Add 1 cc. of N/10 hydrochloric acid to 9 cc. of the dilute solution of the sample (a).

(c) *Dilute inactive solution of the sample.*—Immerse a stoppered glass vessel, containing 45 cc. of the dilute solution of the sample (a) and 5 cc. of N/10 hydrochloric acid. in boiling water for 15 minutes and filter.

(d) *Standard solution containing 0.5 mg. of active U. S. P. pepsin per cc.*—Immerse a stoppered test tube containing 18 cc. of the dilute solution of the sample (a) in boiling water for 10 minutes and, after cooling, add 2 cc. of the standard pepsin solution, containing 5 mg. of pepsin per cc., and filter if necessary.

If the solutions to be tested are not clear, filter through hardened filters. If, however, they can not thus be clarified, make check comparison tubes containing the same amounts of the preparation made up in the same way with 2 cc. of water in place of the ricin solution used in the determination.

43 DETERMINATION.

To each of 15 tubes, add from a burette 2 cc. of the ricin solution and 0.5 cc. of N/10 hydrochloric acid, heat to 37.5°C. and add the following quantities of the solutions:

To the first 5 tubes, add 0.00–1.00 cc. of the dilute comparison solution of the sample **42** (b) in 0.25 cc. increments, and 1.00–0.00 cc. of the dilute inactive solution of the sample **42** (c) in 0.25 cc. decrements. To the next 5 tubes, add 1.00–0.00 cc. of the dilute inactive solution of the sample in 0.25 cc. decrements and 0.00–1.00 cc. of the standard solution containing 0.5 mg. of active U. S. P. pepsin per cc. **42** (d) in 0.25 cc. increments. To the last 5 tubes, add 1.00–0.00 cc. of N/10 hydrochloric acid in 0.25 cc. decrements and 0.00–1.00 cc. of the standard pepsin solution containing 0.5 mg. of pepsin per cc. **42** (b) in 0.25 cc. increments.

By comparing any tube of the first group of 5 with the tubes in the remaining groups the degree of proteolytic activity of the dilute comparison solution of the sample may be matched against known amounts of standard pepsin both in ordinary acid medium, last group of 5, and in the same medium as the sample itself, second group.

Introduce the acid and the dilute inactive solution of the sample into the tubes first and then pour in the solutions to be tested as rapidly as possible from graduated pipettes, noting the total time consumed in the process after adding the pepsin.

After the addition of the solution to be tested, again immerse the test tubes in the 37.5°C. bath, preferably arranged in corresponding order in a partitioned square or oblong wire rack, such as is used in bacteriological work. Shake and examine the tubes from time to time for 1–2 hours, noting the time when the digestion begins and ends. In case of very weak solutions they may be allowed to digest overnight.

If the rate of digestion is the same in each group, the dilute comparison solution of the sample contains exactly 0.5 mg. of pepsin per cc. If the rate is more rapid in the first group than in the others, it is stronger, the comparative strength being closely indicated by the time of action in the tube containing less of the solution. If the rate of clearing is more rapid in the last group than in the second, some interfering substance is present and must be removed by dialysis, or by evaporation in vacuo at a low temperature until, upon re-examination and further dilution or concentration, the rate of digestion is identical or nearly so in each series.

Smaller quantities of pepsin may be determined in the same way by comparing them with more dilute solutions of standard pepsin. Thus 0.05 mg. of U. S. P. pepsin can be readily detected by the nearly complete solvent action on the ricin precipitate in less than 2 hours. A marked action on the ricin within the same time is shown by 0.005 mg. For all practical purposes the absence of an appreciable solvent action after 4 hours' digestion indicates the absence of pepsin. Express the result in per cent, assuming U. S. P. pepsin to be 100 per cent pure and calculating the result according to the dilution found necessary in preparing the dilute solution of the sample.

TURPENTINE.

44 COLOR.—TENTATIVE.

Fill a 200 cc. flat-bottomed colorimeter tube, graduated in mm., to a depth of 40–50 mm. with the turpentine. Place the tube in a colorimeter and place on or under it a No. 2 yellow Lovibond glass. Over or under a second graduated tube in the colorimeter, place a No. 1 yellow Lovibond glass and run in the same turpentine until the color matches as nearly as possible the color in the first tube. Read the difference in depth of the turpentine in the 2 tubes. If this difference is 50 mm. or more, the turpentine is "standard".

45 SPECIFIC GRAVITY.—TENTATIVE.

Determine the specific gravity at $\frac{20^\circ C.}{4^\circ}$ by means of a pycnometer. The specific gravity may also be determined somewhat less accurately at any convenient temperature with a plummet, correcting the result by using the factor 0.00082 for each degree that the temperature of the determination differs from the standard temperature.

46 REFRACTIVE INDEX.—TENTATIVE.

Determine the refractive index at any convenient temperature with an accurate instrument and calculate the result to 20°C., using the correction factor 0.00045 for each degree that the temperature of the determination differs from 20°C.

47 DISTILLATION.—TENTATIVE.

Use an ordinary Engler flask (the internal diameter of the side tube must be 6–7 mm.) and condenser[11] and heat the flask in a glycerin or oil bath[12]. Fit the flask with a thermometer reading 145°–200°C., in such a way that the mercury bulb shall be opposite the side tube of the flask and the 175° mark below the cork. Place 100 cc. of the turpentine in the flask, connect with the condenser and distil. Conduct the distillation so that the distillate passes over at the rate of 2 drops per second. Note the initial distilling temperature and the percentage distilling below 170°C.

POLYMERIZATION.—TENTATIVE.

48 REAGENT.

38N sulphuric acid.—Mix 140 grams of concentrated sulphuric acid with sufficient liquid, fuming sulphuric acid (about 10 grams) to obtain an acid slightly stronger than 38N. Determine the exact strength[13] of this mixture and also of the concentrated acid as follows: Weigh out 6–8 grams in a bulb, having a capillary tube in the lower end and a tube with a stop-cock in the upper end, fitted with a platinum wire for suspending on a balance. (The bulb is filled by the aid of a slight vacuum, and the lower end of the capillary is emptied by closing the stop-cock simultaneously with the withdrawal of the capillary from the acid; after which it is wiped off first with a wet and then with a dry piece of cloth.) Run the acid into cold water, make up to volume and titrate an aliquot of the solution against standard alkali or add an excess of ammonium hydroxid to an aliquot, evaporate to dryness, dry to constant weight at 120°–130°C. and weigh as ammonium sulphate. Calculate the sulphur trioxid content of the acid and add sufficient concentrated sulphuric acid to make it exactly 82.38 per cent of SO_3. The acid must be carefully protected against absorption of water from the air.

49 DETERMINATION.

Place 20 cc. of the 38N sulphuric acid (100.92[4] per cent) in a graduated, narrow-necked Babcock flask, stopper, place in ice water and cool. Add slowly 5 cc. of the turpentine. Mix the contents gradually, cool from time to time and do not allow the temperature to rise above 60°C. When the mixture no longer warms on shaking, agitate thoroughly, place in a water bath and heat to 60°–65°C. for about 10 minutes, keeping the contents of the flask thoroughly mixed by vigorous shaking 5 or 6 times. Cool to room temperature and fill the flask with concentrated sulphuric acid until the unpolymerized oil rises into the graduated neck. Centrifugalize 4–5 minutes at about 1200 revolutions per minute, or allow to stand for 12 hours. Read the unpolymerized residue, notice its consistency and color and determine its refractive index.

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XXIX. SOILS.—TENTATIVE.

1 DIRECTIONS FOR SAMPLING.

Remove from the surface all vegetable material not incorporated with the soil. Take a sufficient number of samples to insure securing a composite sample which will be representative of the tract sampled, to a depth which will include the average depth of plowed soil, usually about 7 inches, and a composite sample from each important and distinctly different soil stratum to the depth of 40 inches, using a soil tube or auger, whichever may be best adapted to the soil conditions. If a soil auger is used, before boring deeper the hole should be enlarged and carefully cleaned out with the soil auger to prevent contamination of the several sub-strata samples while being withdrawn. The sampling should be done when the soil is reasonably dry. Mix the samples of each depth thoroughly and dry in a well-aired, cool place.

It is recommended that the weight of a given volume of the soil as it lies in the field be taken for calculating the percentage results obtained by analysis to pounds per given area of the soil.

In view of the variability characteristic of field soils within small distances, it seems impossible at the present time to devise a perfect method for sampling. The method given above comes as near as any, that may now be adopted, to filling the requirements.

2 PREPARATION OF SAMPLE.

(**a**) Pulverize the air dried soil, using porcelain pebble mill or other equally effective method which will not reduce the rock fragments, to pass through a sieve having circular openings 1 mm. in diameter. Thoroughly mix the sifted material and preserve in a suitable stoppered container. Weigh and discard the detritus.

(**b**) If necessary for determination of total constituents, pulverize more finely a subsample of (**a**).

If for any reason deviations from this procedure are deemed necessary, they should be reported with the results.

3 MOISTURE.

Dry 2 grams of the sample prepared as directed under **2** (**a**) in a wide-mouthed weighing bottle at 100°C. to constant weight. Report the loss of weight as percentage of the moisture free weight of the sample taken.

4 LOSS ON IGNITION.

Ignite soil from **3** in a platinum dish or suitable substitute to full redness, stirring occasionally, until organic matter is destroyed. If the soil contains appreciable quantities of carbonates, moisten, after cooling, with a few drops of a saturated solution of ammonium carbonate; dry and heat to dull redness to expel the ammonium salts; cool in a desiccator and weigh.

The analyst is cautioned that this method gives only a crude approximation of the organic matter and is less accurate in soils containing much colloidal material.

CARBONATE CARBON.

5

APPARATUS.

(**a**) *Quadruplicate shaking apparatus for determination of carbonate carbon in soils of high or low carbonate content*[1].—This consists of a horizontal holder (*H*) 21 inches long, ⅞ inch thick and 1¾ inches wide, with bored holes and entrance thereto made to loosely fit the neck of a 300 cc. Erlenmeyer flask, which takes a No. 6 rubber stopper. Holder (*H*) is suspended from a horizontal bar by means of brass strips 1¾ inches wide and 24 inches long. The common intake for purification of the incoming air leads from a tube about 25 inches long. This tube stands upright, through a rubber stopper, in a 1 liter Erlenmeyer flask and has inserted in the top a large nitrogen distillation bulb, which prevents the mechanical transmission of any of the purifying sodium hydroxid.

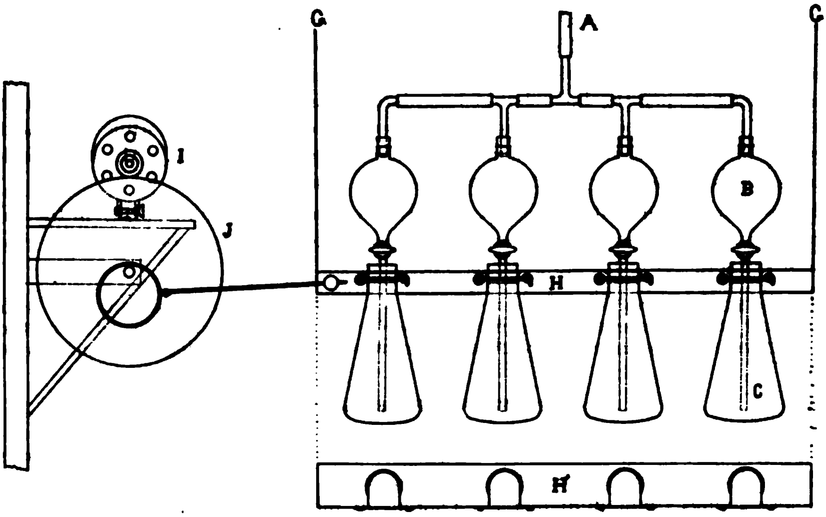

FIG. 15. QUADRUPLICATE SHAKING APPARATUS FOR DETERMINATION OF CARBONATE CARBON CONTENT IN SOILS.

The driving wheel (*J*) is ¾ inch thick and 7 inches in diameter. The eccentric attached to its face is ¾ inch in thickness and 2 inches in diameter. This eccentric is grooved to permit free rotation of the driving shaft which is fastened to the end of the holder (*H*) by means of a binding post. Driving power to effect agitation is obtained by use of the motor (*I*) which is best secured by the use of a sewing machine motor, or by dismantling a small desk fan. If the motor available has no rheostat the speed can be easily controlled by a battery of 4 lamps. The motor is hinged upright on the support so that the pulley will rest upon the edge of the driving wheel. The pulley of the motor is inserted into a rubber stopper, which eliminates noise of friction.

The absorption towers (*D*) are at least 25 inches high and 1 inch in diameter. They contain alternating pockets of solid glass rods and small glass beads resting upon an inverted test tube 2½ inches long. The rubber connection on the intake cock of the tower (*D*) is used to disconnect the glass tube which extends to the rubber connection on the safety bulb tube leading from flask (*C*).

(**b**) *Single apparatus for determination of carbonate carbon in soils high in carbonates*[2].—The apparatus (Fig. 17) consists of an evolution bulb (*J*), a wash bottle (*W*), and an absorption tower (*A*). The evolution bulb (*J*) consists of a 300 cc. dropping funnel with the stem bent as shown. The finely ground soil or other material is placed in this bulb and the acid solution introduced through the funnel (*I*). On aspiration the air enters through (*P*) and rises from the narrow neck of the bulb, effecting an efficient agitation of the soil and solution. A rather wide-mouthed dropping funnel should be selected in order to take at least a No. 3 stopper. The sides of the funnel should slope uniformly to the outlet in the stopper and should not leave a considerable shoulder around the outlet of the stopper on which soil may rest. The wash bottle (*W*) containing silver sulphate is inserted to remove hydrochloric acid when this acid is used to decompose the carbonates.

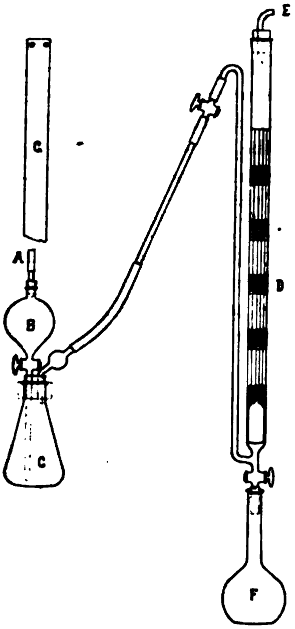

FIG. 16. ABSORPTION TOWER FOR CARBON DIOXID.

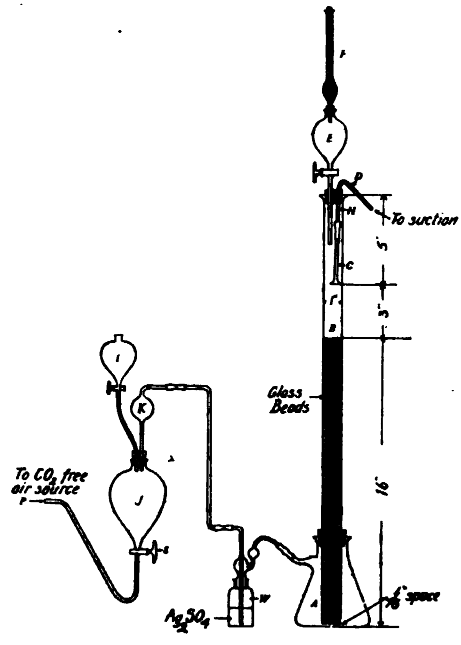

FIG. 17. SINGLE APPARATUS FOR THE DETERMINATION OF CARBONATE CARBON.

The absorption apparatus consists of a 500 cc. suction flask (*A*), a glass tube (*B*), of dimensions as shown, a 50 cc. dropping funnel (*E*), and a soda lime tube (*F*), connected as indicated in the figure. A glass rod (*C*) with a head as shown is attached to the outlet and serves to break up any bubbles that may rise into the tube. The outlet tube has a hole at (*H*).

6 DETERMINATION.

Pulverize the sample to pass a 100 mesh sieve, so as to expose to the action of the liberating acid as much as possible any calcite which may be included in quartz crystals. For soils low in carbonates use a 10, 25, or 50 gram charge in the quadruplicate shaking device described under **5** (**a**). For soils sufficiently high in carbonates to justify 2 or 3 gram charges, the single apparatus described under **5** (**b**) may be used.

Introduce the charges into the 300 cc. evolution flasks, aspirate 5 minutes in order to free the apparatus of atmospheric carbon dioxid, release the suction and introduce 10, 25, or 50 cc. of N/2 sodium or potassium hydroxid solution into the absorption tower. Apply a suction of 5 inches and then introduce 60 cc. of dilute hydrochloric acid (1 to 10) upon the soil contained in the Erlenmeyer flasks, regulating the intake of air by means of screw cocks placed just beyond the absorption towers. Agitate and aspirate for 30 minutes. Then release the suction and draw off the absorbent solution into 500 cc. flasks, washing the towers with a succession of fillings of carbon dioxid-free water, using a minimum volume of 250 cc. Add 10 cc. of a neutral aqueous solution of barium chlorid, made by dissolving 250 grams of barium chlorid ($BaCl_2.2H_2O$) in 1 liter of water. Make to volume and agitate. If the barium carbonate precipitate be light, the titration of the residual hydroxid may be made in the presence of the precipitate. If the barium carbonate be heavy, permit the precipitate to settle and pipette off an aliquot of 200 cc. of the clear supernatant solution; or immediately filter enough to yield a 200 cc. or 400 cc. aliquot, by suction through a 10 cm. Büchner funnel, into a beaker placed under a bell jar. Titrate the excess of hydroxid, using phenolphthalein as an indicator. With a small bore burette, permitting split-drop readings to hundredths of a cc., use N/2 acid for the titration. With larger bore burettes use N/10 or N/4 acid.

If the soil is known to be derived from the limited magnesite area, or if a soil or subsoil be from the glaciated region, where transported dolomite occurs in considerable amounts, the agitation and aspiration should be repeated or continued until the disintegration is completed, if necessary using 1 to 5 hydrochloric acid.

ORGANIC CARBON.

Wet Combustion Method.

7 REAGENTS.

(a) *Oxidizing solution.*—Dissolve 85 grams of chromic anhydrid in 100 cc. of water and dilute to 250 cc. with phosphoric acid, 85 per cent strength.

(b) *Acid solution.*—Mix equal volumes of 85 per cent phosphoric acid and concentrated sulphuric acid.

8 DETERMINATION.

Introduce 1–5 gram charges of soil, depending upon the organic matter content, into each of the four 300 cc. Pyrex Erlenmeyer flasks, 5 (a). Free the apparatus of atmospheric carbon dioxid and then introduce into each absorption tower 25 or 50 cc. of N/2 sodium or potassium hydroxid. Apply suction of 5 inches and run into each Erlenmeyer flask 10 cc. of the oxidizing solution. Then add 25–40 cc. of the acid mixture. Gently agitate the flasks and place a low flame under each. Continue the gentle agitation and heating for 30 minutes subsequent to attaining of the boiling point. Between the Erlenmeyer flasks and the absorption towers, insert a glass bulb of about 1½ inches diameter, and bend the tube leading from this bulb into the Erlenmeyer flask so as to permit the return of the condensed water along the side of the flask. In order to guard against the mechanical aspiration of the liberating acids, place an empty absorbent bead-filled tower between each glass bulb and the tower which contains the hydroxid absorbent.

At the end of the agitation and aspiration, release the suction and wash out the absorbent into a 500 cc. flask. Then precipitate the sodium or potassium carbonate by adding 10 cc. of a neutral aqueous solution of barium chlorid (250 grams of barium chlorid ($BaCl_2.2H_2O$) per liter). Make to volume and permit the precipitate of barium carbonate to settle. Pipette off an aliquot of 200 cc. and titrate the residual hydroxid with N/2 acid, using phenolphthalein as an indicator and splitting drops by the use of a stirring rod near the end of the titration; or obtain the desired aliquot by pipetting from the clear filtrate by throwing the contents of the 500 cc. flask on a 10 cm. Büchner funnel, and filter with suction into a large beaker placed under a bell jar. The difference between the residual hydroxid in terms of N/2 alkali and the N/2 sodium hydroxid originally used is equivalent to the carbon dioxid formed by wet combustion of the organic carbon plus the inorganic carbon dioxid present in the sample. Subtract the amount of carbon dioxid, as determined under **6**, from the total, as determined above. The difference represents the carbon dioxid derived from oxidation of the organic carbon.

Dry Combustion Method.

9

APPARATUS.

(**a**) *A calorimeter bomb.*—Use a type that permits the recovery and transfer of the entire solid residue of the exploded charge to a small vessel by means of a jet of water.

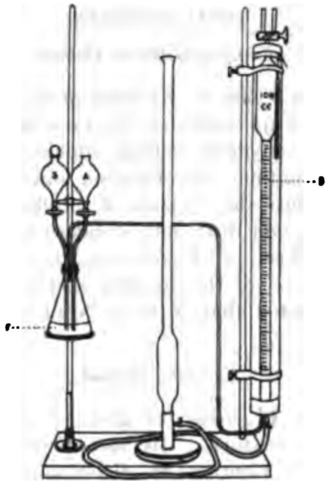

FIG. 18. PARR'S APPARATUS FOR THE DETERMINATION OF CARBON DIOXID.

(**b**) *Parr's apparatus for determining carbon dioxid*[3].—Illustrated in Fig. 18.

This consists of a 150 cc. Erlenmeyer flask (*F*) fitted with a 3-holed stopper through 2 holes of which the stems of 2 dropping funnels (*S*) and (*A*) extend almost to the bottom of the flask. A capillary tube, passing through the third hole and flush with the bottom of the stopper, connects with the gas burette.

(**c**) *A simple Hemple gas pipette.*—Contains potassium hydroxid solution (30 grams of potassium hydroxid dissolved in 70 cc. of water).

10

DETERMINATION.

Introduce 2 grams of soil as prepared under **2** (**a**) (1 gram if high in organic matter), 0.75 gram of magnesium powder, and 10 grams of sodium peroxid, into the closed dry calorimeter bomb, and mix thoroughly by shaking the bomb. Explode the charge by means of an electric spark or by dropping a red hot plug into the bomb through an automatic valve which closes immediately after the plug enters. Remove the residue from the bomb, using as little hot water as possible, heat to boiling, and transfer to the receiving funnel (*S*) of Parr's apparatus. From the acid funnel (*A*) run 50 cc. of sulphuric acid (1 to 2) into the flask (*F*). Connect the apparatus and slowly add the contents from the receiving funnel (*S*). The carbon dioxid generated passes through the capillary tube into the graduated burette. Heat the contents of the flask (*F*) to boiling and boil for 1 minute, then force the gases into the graduated burette by introducing water into the flask (*F*) through the funnel (*S*). Read the burette, recording the temperature and pressure. Pass the gas into the absorption pipette containing potassium hydroxid solution. Shake the gas with the solution until the carbon dioxid is wholly absorbed. Return the residual gas to the graduated burette, and again read the burette noting the temperature and pressure. The difference in readings calculated to standard conditions of temperature and pressure gives the number of cc. of carbon dioxid derived from the total carbon in the sample. Conduct a blank determination upon the reagents used. If an appreciable amount of carbon dioxid is obtained in the blank, the result expressed in terms of total carbon must be corrected accordingly.

Determine the inorganic carbon, as directed under **6**, and subtract it from the total carbon to obtain the organic carbon.

TOTAL NITROGEN.

11

Gunning-Hibbard Method.

Digest 10 grams of soil in a 500 cc. Kjeldahl or flat-bottomed boiling flask with 30–40 cc. of sulphuric acid and approximately 10 grams of salt mixture composed of: 10 parts potassium sulphate or anhydrous sodium sulphate, 1 part ferrous sulphate and ½ part copper sulphate. Continue the digestion until the mixture is colorless or nearly so. After cooling, dilute the contents of the flask with water, add an excess of sodium hydroxid solution and distil into standard acid. The distillation may be carried out in the digestion flask, or, if preferred, the solution may be transferred to a copper flask. Collect 150 cc. of the distillate and titrate the excess of acid with standard alkali. It is suggested that N/10 or N/14 standard solution be used for convenience.

12

Kjeldahl Method.

Proceed as directed under **11**, using 0.7 gram of mercuric oxid or 0.65 gram of mercury instead of the salt mixture. Mix immediately and heat over a low flame, gradually increasing the heat. Continue the digestion until the mixture is colorless or nearly so. After cooling, dilute the contents of the flask, add 25 cc. of potassium sulphid solution (40 grams of potassium sulphid in 1 liter of water) and an excess of alkali, and proceed with the distillation and titration as directed under **11**.

13

SODIUM CARBONATE FUSION OF THE SOIL.

Thoroughly mix on glazed paper 1 gram of soil, ground to an impalpable powder, with 5 grams of sodium carbonate. Transfer carefully to a 40 cc. platinum crucible. Cover, heat at low redness until fusion begins, then increase the heat until a clear, quiet fusion results. Finally, give full heat of Méker burner for 10 minutes, having the flame oblique to insure good oxidation. Pour into a large platinum dish set in water. Place the crucible and cover in a wide 200 cc. beaker. Cover with water, transfer the infused lump from the platinum dish to the beaker and rinse the dish with dilute hydrochloric acid into the beaker. Add 15 cc. of concentrated hydrochloric acid to the contents of the beaker, cover, and place upon a steam bath, until the fused mass has disintegrated. Transfer the mixture to the platinum dish and evaporate to dryness on a steam bath.

14 SILICA.

Take up the residue from **13** in dilute hydrochloric acid (1 to 10) and filter the mixture so obtained (a 9 cm. Büchner funnel with suction may be used advantageously). Wash with hot water containing 1–2 cc. of hydrochloric acid per liter. Collect the filtrate and washings in a dish, preferably a casserole, and dehydrate on a steam bath until the silica assumes a crystalline appearance. Moisten with hydrochloric acid and repeat the dehydration. Add 5 cc. of concentrated hydrochloric acid and 100 cc. of hot water. Mix thoroughly, filter and wash. Add the residue to the main portion of silica obtained from the first filtration. Make up the combined filtrate and washings to 500 cc. and save for subsequent determinations. Place the two silica residues with filters in a porcelain crucible. Ignite slowly at first to burn off filter paper and then with a strong flame, preferably a blast lamp, to constant weight.

15 FERRIC AND ALUMINIC AND TITANIC OXIDS AND PHOSPHORUS.

To an aliquot of the solution from **14** (50 or 100 cc. according to the probable amount of iron present) add ammonium hydroxid, drop by drop, until the precipitate formed requires several seconds to dissolve, thus leaving the solution faintly acid. Heat nearly to the boiling point and add sufficient ammonium hydroxid to precipitate all iron, alumina, etc. Allow to boil in a covered beaker for about 1 minute, remove and if no ammonia is given off (as detected by smelling), add more drop by drop until it can be detected. Do not allow the precipitate to settle, but stir and pour on the filter. Wash immediately with hot water containing ammonium nitrate, using a fine jet which is played around the edge of the precipitate, thus cutting it free from the paper in order to produce rapid filtration. Wash the precipitate several times, return it to the original beaker, dissolve with a few drops of hydrochloric acid and warm. Reprecipitate the iron, alumina and phosphoric acid with ammonium hydroxid as above, filter and wash until free from chlorids. Reserve the filtrate and washings from both the first and second precipitations for the determination of calcium and magnesium.

Dry the precipitate, remove it from the filter, and ignite over a Bunsen flame, the filter being incinerated separately and the residue added to the precipitate. Then ignite to bright redness, cool in a desiccator and weigh as ferric oxid (Fe_2O_3), aluminium oxid (Al_2O_3), titanium oxid (TiO_2), and phosphorus pentoxid (P_2O_5). Transfer this residue to a flask and digest with several cc. of dilute sulphuric acid (1 to 4), heating to accelerate solution. When solution is complete reduce with zinc, cool and determine ferric oxid by titration with N/50 potassium permanganate solution.

Or, in lieu of the above, evaporate 50 or 100 cc. of the solution from **14** with the addition of 10 cc. of sulphuric acid until all hydrochloric acid is expelled, dilute with water, reduce with zinc and determine ferric oxid by titration with N/50 potassium permanganate solution.

Subtracting the ferric oxid, together with the phosphorus pentoxid determined in **21** or **22**, from the collective weight of ferric, aluminic and titanic oxids and phosphorus pentoxid determined above, gives aluminium and titanium oxids.

16 CALCIUM.

Concentrate the combined filtrates and washings from **15** to about 50 cc., cool, add ammonium sulphid to precipitate manganese, filter and wash with hot water. Evaporate the solution to about 50 cc., make slightly alkaline with ammonium hydroxid and add, while still hot, ammonium oxalate solution, drop by drop, so long as any precipitate is produced, adding a few cc. in excess to convert the magnesium also into oxalate. Heat to boiling, allow to stand for 3 hours or longer, decant the clear solution on the filter, pour 15–20 cc. of hot water on the precipitate and again decant the clear solution on the filter. Dissolve the precipitate in the beaker with a few drops of hydrochloric acid, add a little water, and reprecipitate, boiling hot, by adding ammonium hydroxid and a little ammonium oxalate solution. Allow to stand as before and filter through the same filter. Wash free from chlorids with hot water. Reserve the filtrates and washings from both precipitations for the determination of magnesium (**17**). From this point complete the determination by one of the following methods:

(1) Ignite the precipitate and determine the calcium as calcium oxid.

(2) Mix the oxalate precipitate, after incineration of the filter with finely pulverized and dried ammonium sulphate and drive off the excess of the sulphate by careful heating of the upper portion of the crucible. Complete ignition and weigh as calcium sulphate.

(3) Dissolve the calcium oxalate precipitate from the filter by means of dilute sulphuric acid, collecting the solution in the beaker employed for precipitation, and titrate while hot with a standard solution of potassium permanganate. Finally add the filter paper to the solution and complete the titration.

17

MAGNESIUM.

Evaporate the combined filtrates and washings from **16** on the water bath to about 100 cc. and add cautiously 20 cc. of concentrated nitric acid. Evaporate to dryness and heat carefully on a hot plate to remove ammonium salts. Add 5 cc. of hydrochloric acid and evaporate nearly to dryness. Dissolve the residue in hot water and a small amount of hydrochloric acid. If necessary, filter the solution and wash the filter paper with about 100 cc. of hot water. Precipitate the magnesium as magnesium ammonium phosphate by the addition of 3 cc. of a 10 per cent solution of ammonium phosphate and sufficient ammonium hydroxid to make the solution slightly alkaline. Stir the solution vigorously. Allow to stand 15 minutes; add 15 cc. of ammonium hydroxid and allow the precipitation to proceed overnight. Filter, wash the precipitate with 2 per cent ammonium hydroxid solution, transfer to a porcelain crucible, ignite and determine as magnesium pyrophosphate ($Mg_2P_2O_7$). The filtration may be made through a Gooch.

MANGANESE.

18

REAGENTS.

(**a**) *Dilute nitric acid (1 to 4).*—Free from brown oxid of nitrogen by aeration.

(**b**) *Sulphuric acid (1 to 3).*

(**c**) *Dilute sulphuric acid.*—Dilute 25 cc. of concentrated acid to 1 liter with water. Add enough permanganate solution to color faintly the dilute acid.

(**d**) *Standard manganous sulphate solution.*—Dissolve 0.2877 gram of pure potassium permanganate in about 100 cc. of water, acidify the solution with sulphuric acid and heat to boiling. Add slowly a sufficient quantity of dilute solution of oxalic acid to discharge the color. Cool and dilute to 1 liter. One cc. of this solution is equivalent to 0.1 mg. of manganese. The standard should be prepared by following the same procedure as is used for the sample. This solution is more permanent than a solution of potassium permanganate, which may, however, be used. To prepare it dissolve 0.288 gram of potassium permanganate in water and dilute the solution to 1 liter.

(**e**) *Sodium bismuthate.*—Pure dry salt.

19

DETERMINATION.

Treat 1 gram of the original soil with hydrofluoric and sulphuric acids. Evaporate to dryness, ignite and fuse the residue with potassium pyrosulphate. Dissolve in water, add nitric acid and evaporate to dryness. Again dissolve in water, add 25 cc. of dilute nitric acid (1 to 3), add about 0.5 gram of sodium bismuthate, and heat until the permanganate color disappears. Add a few drops of a solution of ammonium or sodium bisulphate to clear the solution and again boil to expel oxids of nitrogen. Remove from the source of heat, cool to 20°C., again add 0.5 gram of sodium bismuthate, and stir. When the maximum permanganate color has developed, filter through an alundum or Gooch crucible containing an asbestos mat ignited and washed with potassium permanganate. Wash the precipitate with dilute sulphuric acid until the washings are colorless. Transfer the filtrate to a colorimeter tube and compare the color of it with that of standards prepared from the potassium permanganate solution. To prepare the standards, dilute with sulphuric acid (solution C) portions of 0.2, 0.4, 0.6 cc., etc., of the permanganate solution to the same volume as the filtrate.

20 SULPHUR.

Evaporate 100 or 200 cc. of the solution from **14** nearly to dryness on a water bath to expel the excess of acid, add 400 cc. of water, heat to boiling and add, drop by drop, a 10 per cent hot barium chlorid solution until no further precipitation occurs. Continue near the boiling point for about 5 minutes, allow to stand for 5 hours or longer in a warm place, decant the liquid on a tared Gooch or on an ashless filter, treat the precipitate with 15–20 cc. of boiling water, transfer to the filter, and wash free from chlorids with boiling water. Dry the filter, ignite over a Bunsen burner, and weigh as barium sulphate.

For the determination of sulphur as sulphates agitate soil with water for 7 hours, proceeding otherwise as directed under **30**.

PHOSPHORUS.

21 *Sodium Peroxid Method.*

Place 10 grams of sodium peroxid in an iron or porcelain crucible and thoroughly mix with it 5 grams of the soil. If the soil has very little organic matter, add a little starch to hasten the action. Heat the mixture carefully by applying the flame of a Bunsen burner directly upon the surface of the charge and the sides of the crucible until the action starts. Cover crucible until reaction is over and keep at a low red heat for 15 minutes. Do not allow fusion to take place. By means of a large funnel and a stream of hot water, transfer the charge to a 500 cc. volumetric flask, acidify with hydrochloric acid and boil. Cool and make up to the mark. If the action has taken place properly there should be no particles of undecomposed soil in the bottom of the flask. Allow the silica to settle and draw off 200 cc. of the clear solution.

Precipitate the iron, aluminium, and phosphorus with ammonium hydroxid; filter, wash several times with hot water, return the precipitate to the beaker, and dissolve the precipitate in hot hydrochloric acid, pouring the acid upon the filter to dissolve any precipitate remaining. Evaporate the solution and washings to complete dryness on a water bath. Take up with dilute hydrochloric acid, heating if necessary, and remove the silica by filtration. Evaporate filtrate and washings to about 10 cc., add 2 cc. of concentrated nitric acid, and neutralize with ammonium hydroxid. Add nitric acid, until the solution is clear, avoiding an excess. Heat at 40°–50°C. on a water bath, add 15 cc. of molybdate solution, keeping at this temperature for 1–2 hours. Let stand overnight, filter, and wash free from acid with 0.1 per cent solution of ammonium nitrate, and finally once or twice with cold water. Transfer filter to beaker and dissolve in standard potassium hydroxid (1 cc. = 0.5 mg. phosphorus pentoxid). Titrate the excess of potassium hydroxid with standard nitric acid, using phenolphthalein as an indicator. Or, after adding the 15 cc. of molybdate solution, allow to stand for 3 hours at a temperature not above 60°C., filter on a small filter or on a Gooch crucible and wash with cold water until 2 fillings of the filter do not greatly diminish the color produced with phenolphthalein by 1 drop of standard alkali. Return the filter and precipitate to the same beaker used for precipitating the phosphomolybdate, dissolve the yellow precipitate in standard sodium or potassium hydroxid, add a few drops of phenolphthalein solution and titrate excess of alkali with standard acid. One cc. of the standard alkali is equivalent to 0.0005 gram of phosphorus pentoxid (P_2O_5).

22 *Magnesium Nitrate Method.*

Place 5 grams of soil in a porcelain dish. Moisten with 5–7 cc. of magnesium nitrate solution. (Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess, boil, filter from the excess of magnesia, ferric oxid, etc., and dilute to 2 liters.) Dry on a water bath and burn off the organic matter at low redness. Cool, moisten slightly with water, add 10 cc. of concentrated hydrochloric acid, and digest for 2 hours on a water bath, keeping the dish covered with a watch glass and stirring 2 or 3 times during digestion. Make up to 250 cc., mix well, and transfer to a dry folded filter, pouring back on the filter until the solution runs through clear. Make the determination on aliquots corresponding to 2 or 4 grams of the soil, depending upon the amount of phosphorus present. Dry, take up with hydrochloric acid and water and filter, the filtrate and washings not exceeding 30–40 cc. Make alkaline with ammonium hydroxid, and dissolve the precipitate with concentrated nitric acid, using a slight excess. Add gradually, while shaking, 5–15 cc. of molybdate solution, shaking thoroughly. Keep the solution at 40°–50°C. for an hour, let stand overnight at room temperature, filter and wash well with cold water. Return filter and precipitate to the same flask and determine phosphorus volumetrically, as directed under 21.

23 POTASSIUM AND SODIUM[4].

Triturate gently 0.5 or 1 gram of the finely ground soil with 1 gram of dry ammonium chlorid in a smooth mortar, then add 8 parts of calcium carbonate and mix intimately. Transfer the mixture to a platinum crucible, rinsing the mortar with a little calcium carbonate. Heat the crucible gradually until fumes of ammonium salts no longer appear, and continue until the lower three-fourths only of the crucible are brought to a red heat. Maintain this temperature 40–60 minutes. The temperature should be sufficient to keep the calcium chlorid formed by the reaction of ammonium chlorid with calcium carbonate in a state of fusion. The mass, however, does not become liquid since the fused calcium chlorid is absorbed by the large quantity of calcium carbonate present. If the silicate is fused by the application of too strong heat, disintegration of the mass at the end of the operation with water can not be effected. Moreover, too high a temperature causes volatilization of alkali chlorids. The mass contracts in volume during the ignition, and is usually easily detached from the crucible. Transfer the fused mass to a porcelain dish, slake with hot water, and grind thoroughly with an agate pestle. After washing 5 times by decantation with hot water, transfer to a filter and wash well, 300 cc. of wash water being sufficient. To the filtrate add ammonium carbonate solution to precipitate the calcium and any magnesium present. Allow to settle, decant the supernatant liquid into a porcelain (or platinum) dish and concentrate by evaporation, finally transferring the precipitate to the dish. When the volume is reduced to about 30 cc., add a little ammonium carbonate and ammonium hydroxid, heat, filter into a porcelain (or platinum) dish, evaporate the filtrate to dryness on a water bath and expel ammonium salts by ignition. Dissolve the residual alkali chlorids in 3–5 cc. of water; a little black or brown flocculent matter usually remains undissolved, while the solution may also contain traces of calcium. Add 2–3 drops of ammonium carbonate solution and ammonium hydroxid, warm and filter through a small filter into a weighed platinum dish. Evaporate to dryness on a water bath, carefully heat the residual alkali chlorids to incipient fusion, cool and weigh as sodium and potassium chlorids. Dissolve the combined chlorids in 30 cc. of water, add 1.5 cc. of platinic chlorid solution (10 cc. contains 1 gram of platinum) evaporate to a sirupy consistency, and add 15 cc. of 2,25 N acidulated alcohol (prepared by passing hydrochloric acid gas into a mixture of 2000 cc. of 95 per cent alcohol and 152 cc. of hydrochloric acid, sp. gr. 1.20). Filter through a small filter, wash free from platinum salts with 80 per cent alcohol, then with ammonium chlorid solution (100 grams of ammonium chlorid in 500 cc. of water saturated with potassium platinic chlorid), and finally with 80 per cent alcohol. Dry the precipitate on the filter, dissolve, and wash the precipitate through the filter with hot water into a weighed platinum dish, using suction. Evaporate to dryness, heat in a drying oven for an hour at 120°C., cool in a desiccator, weigh and calculate to potassium oxid (K_2O). Calculate the potassium oxid to potassium chlorid and deduct from the weight of combined sodium and potassium chlorids to obtain sodium chlorid.

24 QUALITATIVE TEST FOR SOIL REACTION-LITMUS PAPER.

Place approximately 50 grams of air-dry soil in a small porcelain evaporating dish freshly rinsed with water. Add water and mix to a thick paste. By means of forceps, lay upon this a strip of the best grade litmus paper. Press firmly against the soil by means of a clean spatula or glass rod, keeping the upper surface of the paper free from soil. Protect and permit to stand for 30 minutes, then note the color of the litmus paper.

NITRATE NITROGEN.

25 PREPARATION OF SOLUTION.

Place 100 grams of air dry soil in a mortar or porcelain evaporating dish, add with constant stirring 1 gram of lime and 200 cc. of water. Allow to settle for 10–20 minutes and filter, making sure that a clear filtrate is obtained. If the filtrate contains 6 parts of chlorin per million or less, proceed as in **27**; if it contains more than 6 parts of chlorin per million, proceed as in **29**.

Method for Soil Solutions Containing Little Chlorin.

26 REAGENTS.

(**a**) *Phenoldisulphonic acid solution.*—Dissolve 25 grams of pure white phenol in 150 cc. of concentrated sulphuric acid, add 75 cc. of fuming sulphuric acid (13–15 per cent SO_3) and heat at 100°C. for 2 hours.

(**b**) *Standard nitrate solution.*—Dissolve 0.722 gram of pure potassium nitrate in 1 liter of nitrate-free water. Evaporate 50 cc. of this solution to dryness in a porcelain dish; treat with 2 cc. of the phenoldisulphonic acid solution, rubbing with a glass rod to insure intimate contact. Dilute to 500 cc. One cc. is equivalent to 0.01 mg. of nitrogen as nitrate. This solution is permanent. Standards for comparison are prepared by adding ammonium hydroxid to measured volumes of it in 100 cc. Nessler tubes.

27 DETERMINATION.

Evaporate 25 cc. of the solution, prepared as directed under **25**, on a water bath. Cool, and add 2 cc. of the phenoldisulphonic acid solution. Triturate thoroughly with a clean glass rod, add 25 cc. of water and strong ammonium hydroxid or potassium hydroxid, drop by drop, with constant stirring, until a permanent yellow color is obtained. Compare the solution in a colorimeter with a standard solution prepared in a similar manner to the unknown. Record as nitrogen in the form of nitrate.

Method for Soil Solutions Containing Considerable Chlorin.

28 REAGENTS.

(**a**) *Aluminium foil.*—This reagent should be the purest obtainable. Cut into strips about 10 cm. long, weighing about 0.5 gram.

(**b**) *Sodium or potassium hydroxid solution.*—Dissolve 250 grams of the purest hydroxid obtainable in 1250 cc. of water. Add 2 or 3 strips of aluminium foil, (**a**), and let stand about 12 hours. Concentrate the solution to 1 liter by boiling.

29 DETERMINATION.

Place 25 cc. of soil solution, prepared as directed under **25**, or such a quantity as contains 0.1 milligram or less of nitrogen in the form of nitrate, in a 300 cc. casserole. Add 2 cc. of the sodium hydroxid solution and concentrate by boiling to about one-third the original volume. Transfer to a 100 cc. test tube, using nitrogen-free water, diluting, if necessary, to a volume of about 75 cc. Prepare a blank (preferably several blanks, since the nitrogen impurity in aluminium is often distributed unevenly) by placing about 75 cc. of nitrogen-free water and 2 cc. of the sodium hydroxid in a 100 cc. test tube. Place a strip of aluminium foil in each tube. Close the mouths of the test tubes with rubber stoppers connected by means of bent glass tubes with other test tubes containing about 50 cc. of slightly acidified ammonia-free water. Allow sample and blank to stand at room temperature for 12 hours or until reduction is complete. Nesslerize the traps. If high in ammonia, indicated by frothing over of the sample, the determination should be discarded. If the traps contain the equivalent of only 1–2 cc. of standard ammonia solution each, they should be disregarded. Transfer sample and blank to distilling flasks using 250 cc. of ammonia-free water for each distil, nesslerize and compare with standards. Subtract the amount of nitrogen in the blank from that found in the sample. Calculate the mg. per liter of nitrogen in the form of nitrate.

30 ALKALI SALTS.

To 100 grams of soil in a 500 cc. bottle, add 250 cc. of water. Stopper, shake thoroughly and allow to stand overnight. Filter through a Pasteur-Chamberland filter. Evaporate 50 cc. of the filtrate in a platinum dish on a steam bath to dryness. Ignite at a low red heat to drive off organic matter. Cool in a desiccator and weigh for total salts. Then dissolve the residue in the platinum dish in 10–15 cc. of hot water. Make up to 40 cc. in a graduated cylinder. Take 10 cc. for titration with N/10 silver nitrate; 10 cc. for titration with N/10 hydrochloric acid for sodium carbonate. Determine sulphates by difference. When much gypsum is present, the solution of the salts in hot water must be filtered through a small filter, the gypsum weighed separately, and subtracted from the total amount of sulphates.

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[2] J. Ind. Eng. Chem., 1915, 7: 1045; 1916, 8: 341.

[3] J. Am. Chem. Soc., 1904, 26: 294, 1640.

[4] Fresenius. Quantitative Chemical Analysis. Revised and Amplified Translation of the 6th German Ed. 1906, 2: 1175; Crookes. Select Methods in Chemical Analysis. 4th ed., 1905, 23; Wiley. Principles and Practice of Agricultural Analysis, 1906, 1: 423; U. S. Geological Survey, Bulletin 422, p. 171.

XXX. REFERENCE TABLES.

1

Munson and Walker's Table.

For calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 gram and 2 grams total sugar), lactose (two forms), and maltose (anhydrous and crystallized).

[Expressed in milligrams.]

Cuprous oxid (Cu_2O)	Copper (Cu)	Dextrose (*d*-glucose)	Invert sugar	Invert sugar and sucrose		Lactose		Maltose		Cuprous oxid (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	
10	8.9	4.0	4.5	1.6	----	3.8	4.0	5.9	6.2	10
11	9.8	4.5	5.0	2.1	----	4.5	4.7	6.7	7.0	11
12	10.7	4.9	5.4	2.5	----	5.1	5.4	7.5	7.9	12
13	11.5	5.3	5.8	3.0	----	5.8	6.1	8.3	8.7	13
14	12.4	5.7	6.3	3.4	----	6.4	6.8	9.1	9.5	14
15	13.3	6.2	6.7	3.9	----	7.1	7.5	9.9	10.4	15
16	14.2	6.6	7.2	4.3	----	7.8	8.2	10.6	11.2	16
17	15.1	7.0	7.6	4.8	----	8.4	8.9	11.4	12.0	17
18	16.0	7.5	8.1	5.2	----	9.1	9.5	12.2	12.9	18
19	16.9	7.9	8.5	5.7	----	9.7	10.2	13.0	13.7	19
20	17.8	8.3	8.9	6.1	----	10.4	10.9	13.8	14.6	20
21	18.7	8.7	9.4	6.6	----	11.0	11.6	14.6	15.4	21
22	19.5	9.2	9.8	7.0	----	11.7	12.3	15.4	16.2	22
23	20.4	9.6	10.3	7.5	----	12.3	13.0	16.2	17.1	23
24	21.3	10.0	10.7	7.9	----	13.0	13.7	17.0	17.9	24
25	22.2	10.5	11.2	8.4	----	13.7	14.4	17.8	18.7	25
26	23.1	10.9	11.6	8.8	----	14.3	15.1	18.6	19.6	26
27	24.0	11.3	12.0	9.3	----	15.0	15.8	19.4	20.4	27
28	24.9	11.8	12.5	9.7	----	15.6	16.5	20.2	21.2	28
29	25.8	12.2	12.9	10.2	----	16.3	17.1	21.0	22.1	29
30	26.6	12.6	13.4	10.7	4.3	16.9	17.8	21.8	22.9	30
31	27.5	13.1	13.8	11.1	4.7	17.6	18.5	22.6	23.7	31
32	28.4	13.5	14.3	11.6	5.2	18.3	19.2	23.3	24.6	32
33	29.3	13.9	14.7	12.0	5.6	18.9	19.9	24.1	25.4	33
34	30.2	14.3	15.2	12.5	6.1	19.6	20.6	24.9	26.2	34
35	31.1	14.8	15.6	12.9	6.5	20.2	21.3	25.7	27.1	35
36	32.0	15.2	16.1	13.4	7.0	20.9	22.0	26.5	27.9	36
37	32.9	15.6	16.5	13.8	7.4	21.5	22.7	27.3	28.7	37
38	33.8	16.1	16.9	14.3	7.9	22.2	23.4	28.1	29.6	38
39	34.6	16.5	17.4	14.7	8.4	22.8	24.1	28.9	30.4	39
40	35.5	16.9	17.8	15.2	8.8	23.5	24.8	29.7	31.3	40
41	36.4	17.4	18.3	15.6	9.3	24.2	25.4	30.5	32.1	41
42	37.3	17.8	18.7	16.1	9.7	24.8	26.1	31.3	32.9	42
43	38.2	18.2	19.2	16.6	10.2	25.5	26.8	32.1	33.8	43
44	39.1	18.7	19.6	17.0	10.7	26.1	27.5	32.9	34.6	44

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MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	
45	40.0	19.1	20.1	17.5	11.1	26.8	28.2	33.7	35.4	45
46	40.9	19.6	20.5	17.9	11.6	27.4	28.9	34.4	36.3	46
47	41.7	20.0	21.0	18.4	12.0	28.1	29.6	35.2	37.1	47
48	42.6	20.4	21.4	18.8	12.5	28.7	30.3	36.0	37.9	48
49	43.5	20.9	21.9	19.3	12.9	29.4	31.0	36.8	38.8	49
50	44.4	21.3	22.3	19.7	13.4	30.1	31.7	37.6	39.6	50
51	45.3	21.7	22.8	20.2	13.9	30.7	32.4	38.4	40.4	51
52	46.2	22.2	23.2	20.7	14.3	31.4	33.0	39.2	41.3	52
53	47.1	22.6	23.7	21.1	14.8	32.1	33.7	40.0	42.1	53
54	48.0	23.0	24.1	21.6	15.2	32.7	34.4	40.8	42.9	54
55	48.9	23.5	24.6	22.0	15.7	33.4	35.1	41.6	43.8	55
56	49.7	23.9	25.0	22.5	16.2	34.0	35.8	42.4	44.6	56
57	50.6	24.3	25.5	22.9	16.6	34.7	36.5	43.2	45.4	57
58	51.5	24.8	25.9	23.4	17.1	35.4	37.2	44.0	46.3	58
59	52.4	25.2	26.4	23.9	17.5	36.0	37.9	44.8	47.1	59
60	53.3	25.6	26.8	24.3	18.0	36.7	38.6	45.6	48.0	60
61	54.2	26.1	27.3	24.8	18.5	37.3	39.3	46.3	48.8	61
62	55.1	26.5	27.7	25.2	18.9	38.0	40.0	47.1	49.6	62
63	56.0	27.0	28.2	25.7	19.4	38.6	40.7	47.9	50.5	63
64	56.8	27.4	28.6	26.2	19.8	39.3	41.4	48.7	51.3	64
65	57.7	27.8	29.1	26.6	20.3	40.0	42.1	49.5	52.1	65
66	58.6	28.3	29.5	27.1	20.8	40.6	42.8	50.3	53.0	66
67	59.5	28.7	30.0	27.5	21.2	41.3	43.5	51.1	53.8	67
68	60.4	29.2	30.4	28.0	21.7	41.9	44.2	51.9	54.6	68
69	61.3	29.6	30.9	28.5	22.2	42.6	44.8	52.7	55.5	69
70	62.2	30.0	31.3	28.9	22.6	43.3	45.5	53.5	56.3	70
71	63.1	30.5	31.8	29.4	23.1	43.9	46.2	54.3	57.1	71
72	64.0	30.9	32.3	29.8	23.5	44.6	46.9	55.1	58.0	72
73	64.8	31.4	32.7	30.3	24.0	45.2	47.6	55.9	58.8	73
74	65.7	31.8	33.2	30.8	24.5	45.9	48.3	56.7	59.6	74
75	66.6	32.2	33.6	31.2	24.9	46.6	49.0	57.5	60.5	75
76	67.5	32.7	34.1	31.7	25.4	47.2	49.7	58.2	61.3	76
77	68.4	33.1	34.5	32.1	25.9	47.9	50.4	59.0	62.1	77
78	69.3	33.6	35.0	32.6	26.3	48.5	51.1	59.8	63.0	78
79	70.2	34.0	35.4	33.1	26.8	49.2	51.8	60.6	63.8	79
80	71.1	34.4	35.9	33.5	27.3	49.9	52.5	61.4	64.6	80
81	71.9	34.9	36.3	34.0	27.7	50.5	53.2	62.2	65.5	81
82	72.8	35.3	36.8	34.5	28.2	51.2	53.9	63.0	66.3	82
83	73.7	35.8	37.3	34.9	28.6	51.8	54.6	63.8	67.1	83
84	74.6	36.2	37.7	35.4	29.1	52.5	55.3	64.6	68.0	84
85	75.5	36.7	38.2	35.8	29.6	53.1	56.0	65.4	68.8	85
86	76.4	37.1	38.6	36.3	30.0	53.8	56.6	66.2	69.7	86
87	77.3	37.5	39.1	36.8	30.5	54.5	57.3	67.0	70.5	87
88	78.2	38.0	39.5	37.2	31.0	55.1	58.0	67.8	71.3	88
89	79.1	38.4	40.0	37.7	31.4	55.8	58.7	68.5	72.2	89

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MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	
90	79.9	38.9	40.4	38.2	31.9	56.4	59.4	69.3	73.0	90
91	80.8	39.3	40.9	38.6	32.4	57.1	60.1	70.1	73.8	91
92	81.7	39.8	41.4	39.1	32.8	57.8	60.8	70.9	74.7	92
93	82.6	40.2	41.8	39.6	33.3	58.4	61.5	71.7	75.5	93
94	83.5	40.6	42.3	40.0	33.8	59.1	62.2	72.5	76.3	94
95	84.4	41.1	42.7	40.5	34.2	59.7	62.9	73.3	77.2	95
96	85.3	41.5	43.2	41.0	34.7	60.4	63.6	74.1	78.0	96
97	86.2	42.0	43.7	41.4	35.2	61.1	64.3	74.9	78.8	97
98	87.1	42.4	44.1	41.9	35.6	61.7	65.0	75.7	79.7	98
99	87.9	42.9	44.6	42.4	36.1	62.4	65.7	76.5	80.5	99
100	88.8	43.3	45.0	42.8	36.6	63.0	66.4	77.3	81.3	100
101	89.7	43.8	45.5	43.3	37.0	63.7	67.1	78.1	82.2	101
102	90.6	44.2	46.0	43.8	37.5	64.4	67.8	78.8	83.0	102
103	91.5	44.7	46.4	44.2	38.0	65.0	68.5	79.6	83.8	103
104	92.4	45.1	46.9	44.7	38.5	65.7	69.1	80.4	84.7	104
105	93.3	45.5	47.3	45.2	38.9	66.4	69.8	81.2	85.5	105
106	94.2	46.0	47.8	45.6	39.4	67.0	70.5	82.0	86.3	106
107	95.0	46.4	48.3	46.1	39.9	67.7	71.2	82.8	87.2	107
108	95.9	46.9	48.7	46.6	40.3	68.3	71.9	83.6	88.0	108
109	96.8	47.3	49.2	47.0	40.8	69.0	72.6	84.4	88.8	109
110	97.7	47.8	49.6	47.5	41.3	69.7	73.3	85.2	89.7	110
111	98.6	48.2	50.1	48.0	41.7	70.3	74.0	86.0	90.5	111
112	99.5	48.7	50.6	48.4	42.2	71.0	74.7	86.8	91.3	112
113	100.4	49.1	51.0	48.9	42.7	71.6	75.4	87.6	92.2	113
114	101.3	49.6	51.5	49.4	43.2	72.3	76.1	88.4	93.0	114
115	102.2	50.0	51.9	49.8	43.6	73.0	76.8	89.2	93.9	115
116	103.0	50.5	52.4	50.3	44.1	73.6	77.5	90.0	94.7	116
117	103.9	50.9	52.9	50.8	44.6	74.3	78.2	90.7	95.5	117
118	104.8	51.4	53.3	51.2	45.0	75.0	78.9	91.5	96.4	118
119	105.7	51.8	53.8	51.7	45.5	75.6	79.6	92.3	97.2	119
120	106.6	52.3	54.3	52.2	46.0	76.3	80.3	93.1	98.0	120
121	107.5	52.7	54.7	52.7	46.5	76.9	81.0	93.9	98.9	121
122	108.4	53.2	55.2	53.1	46.9	77.6	81.7	94.7	99.7	122
123	109.3	53.6	55.7	53.6	47.4	78.3	82.4	95.5	100.5	123
124	110.1	54.1	56.1	54.1	47.9	78.9	83.1	96.3	101.4	124
125	111.0	54.5	56.6	54.5	48.3	79.6	83.8	97.1	102.2	125
126	111.9	55.0	57.0	55.0	48.8	80.3	84.5	97.9	103.0	126
127	112.8	55.4	57.5	55.5	49.3	80.9	85.2	98.7	103.9	127
128	113.7	55.9	58.0	55.9	49.8	81.6	85.9	99.4	104.7	128
129	114.6	56.3	58.4	56.4	50.2	82.2	86.6	100.2	105.5	129
130	115.5	56.8	58.9	56.9	50.7	82.9	87.3	101.0	106.4	130
131	116.4	57.2	59.4	57.4	51.2	83.6	88.0	101.8	107.2	131
132	117.3	57.7	59.8	57.8	51.7	84.2	88.7	102.6	108.0	132
133	118.1	58.1	60.3	58.3	52.1	84.9	89.4	103.4	108.9	133
134	119.0	58.6	60.8	58.8	52.6	85.5	90.1	104.2	109.7	134

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MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (*d*-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	
135	119.9	59.0	61.2	59.3	53.1	86.2	90.8	105.0	110.5	135
136	120.8	59.5	61.7	59.7	53.6	86.9	91.5	105.8	111.4	136
137	121.7	60.0	62.2	60.2	54.0	87.5	92.1	106.6	112.2	137
138	122.6	60.4	62.6	60.7	54.5	88.2	92.8	107.4	113.0	138
139	123.5	60.9	63.1	61.2	55.0	88.9	93.5	108.2	113.9	139
140	124.4	61.3	63.6	61.6	55.5	89.5	94.2	109.0	114.7	140
141	125.2	61.8	64.0	62.1	55.9	90.2	94.9	109.8	115.5	141
142	126.1	62.2	64.5	62.6	56.4	90.8	95.6	110.5	116.4	142
143	127.0	62.7	65.0	63.1	56.9	91.5	96.3	111.3	117.2	143
144	127.9	63.1	65.4	63.5	57.4	92.2	97.0	112.1	118.0	144
145	128.8	63.6	65.9	64.0	57.8	92.8	97.7	112.9	118.9	145
146	129.7	64.0	66.4	64.5	58.3	93.5	98.4	113.7	119.7	146
147	130.6	64.5	66.9	65.0	58.8	94.2	99.1	114.5	120.5	147
148	131.5	65.0	67.3	65.4	59.3	94.8	99.8	115.3	121.4	148
149	132.4	65.4	67.8	65.9	59.7	95.5	100.5	116.1	122.2	149
150	133.2	65.9	68.3	66.4	60.2	96.1	101.2	116.9	123.0	150
151	134.1	66.3	68.7	66.9	60.7	96.8	101.9	117.7	123.9	151
152	135.0	66.8	69.2	67.3	61.2	97.5	102.6	118.5	124.7	152
153	135.9	67.2	69.7	67.8	61.7	98.1	103.3	119.3	125.5	153
154	136.8	67.7	70.1	68.3	62.1	98.8	104.0	120.0	126.4	154
155	137.7	68.2	70.6	68.8	62.6	99.5	104.7	120.8	127.2	155
156	138.6	68.6	71.1	69.2	63.1	100.1	105.4	121.6	128.0	156
157	139.5	69.1	71.6	69.7	63.6	100.8	106.1	122.4	128.9	157
158	140.3	69.5	72.0	70.2	64.1	101.5	106.8	123.2	129.7	158
159	141.2	70.0	72.5	70.7	64.5	102.1	107.5	124.0	130.5	159
160	142.1	70.4	73.0	71.2	65.0	102.8	108.2	124.8	131.4	160
161	143.0	70.9	73.4	71.6	65.5	103.4	108.9	125.6	132.2	161
162	143.9	71.4	73.9	72.1	66.0	104.1	109.6	126.4	133.0	162
163	144.8	71.8	74.4	72.6	66.5	104.8	110.3	127.2	133.9	163
164	145.7	72.3	74.9	73.1	66.9	105.4	111.0	128.0	134.7	164
165	146.6	72.8	75.3	73.6	67.4	106.1	111.7	128.8	135.5	165
166	147.5	73.2	75.8	74.0	67.9	106.8	112.4	129.6	136.4	166
167	148.3	73.7	76.3	74.5	68.4	107.4	113.1	130.3	137.2	167
168	149.2	74.1	76.8	75.0	68.9	108.1	113.8	131.1	138.0	168
169	150.1	74.6	77.2	75.5	69.3	108.8	114.5	131.9	138.9	169
170	151.0	75.1	77.7	76.0	69.8	109.4	115.2	132.7	139.7	170
171	151.9	75.5	78.2	76.4	70.3	110.1	115.9	133.5	140.5	171
172	152.8	76.0	78.7	76.9	70.8	110.8	116.6	134.3	141.4	172
173	153.7	76.4	79.1	77.4	71.3	111.4	117.3	135.1	142.2	173
174	154.6	76.9	79.6	77.9	71.7	112.1	118.0	135.9	143.0	174
175	155.5	77.4	80.1	78.4	72.2	112.8	118.7	136.7	143.9	175
176	156.3	77.8	80.6	78.8	72.7	113.4	119.4	137.5	144.7	176
177	157.2	78.3	81.0	79.3	73.2	114.1	120.1	138.3	145.5	177
178	158.1	78.8	81.5	79.8	73.7	114.8	120.8	139.1	146.4	178
179	159.0	79.2	82.0	80.3	74.2	115.4	121.5	139.8	147.2	179

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MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (*d*-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	
180	159.9	79.7	82.5	80.8	74.6	116.1	122.2	140.6	148.0	180
181	160.8	80.1	82.9	81.3	75.1	116.7	122.9	141.4	148.9	181
182	161.7	80.6	83.4	81.7	75.6	117.4	123.6	142.2	149.7	182
183	162.6	81.1	83.9	82.2	76.1	118.1	124.3	143.0	150.5	183
184	163.4	81.5	84.4	82.7	76.6	118.7	125.0	143.8	151.4	184
185	164.3	82.0	84.9	83.2	77.1	119.4	125.7	144.6	152.2	185
186	165.2	82.5	85.3	83.7	77.6	120.1	126.4	145.4	153.0	186
187	166.1	82.9	85.8	84.2	78.0	120.7	127.1	146.2	153.9	187
188	167.0	83.4	86.3	84.6	78.5	121.4	127.8	147.0	154.7	188
189	167.9	83.9	86.8	85.1	79.0	122.1	128.5	147.8	155.5	189
190	168.8	84.3	87.2	85.6	79.5	122.7	129.2	148.6	156.4	190
191	169.7	84.8	87.7	86.1	80.0	123.4	129.9	149.3	157.2	191
192	170.5	85.3	88.2	86.6	80.5	124.1	130.6	150.1	158.0	192
193	171.4	85.7	88.7	87.1	81.0	124.7	131.3	150.9	158.9	193
194	172.3	86.2	89.2	87.6	81.4	125.4	132.0	151.7	159.7	194
195	173.2	86.7	89.6	88.0	81.9	126.1	132.7	152.5	160.5	195
196	174.1	87.1	90.1	88.5	82.4	126.7	133.4	153.3	161.4	196
197	175.0	87.6	90.6	89.0	82.9	127.4	134.1	154.1	162.2	197
198	175.9	88.1	91.1	89.5	83.4	128.1	134.8	154.9	163.0	198
199	176.8	88.5	91.6	90.0	83.9	128.7	135.5	155.7	163.9	199
200	177.7	89.0	92.0	90.5	84.4	129.4	136.2	156.5	164.7	200
201	178.5	89.5	92.5	91.0	84.8	130.0	136.9	157.3	165.5	201
202	179.4	89.9	93.0	91.4	85.3	130.7	137.6	158.1	166.4	202
203	180.3	90.4	93.5	91.9	85.8	131.4	138.3	158.8	167.2	203
204	181.2	90.9	94.0	92.4	86.3	132.0	139.0	159.6	168.0	204
205	182.1	91.4	94.5	92.9	86.8	132.7	139.7	160.4	168.9	205
206	183.0	91.8	94.9	93.4	87.3	133.4	140.4	161.2	169.7	206
207	183.9	92.3	95.4	93.9	87.8	134.0	141.1	162.0	170.5	207
208	184.8	92.8	95.9	94.4	88.3	134.7	141.8	162.8	171.4	208
209	185.6	93.2	96.4	94.9	88.8	135.4	142.5	163.6	172.2	209
210	186.5	93.7	96.9	95.4	89.2	136.0	143.2	164.4	173.0	210
211	187.4	94.2	97.4	95.8	89.7	136.7	143.9	165.2	173.8	211
212	188.3	94.6	97.8	96.3	90.2	137.4	144.6	166.0	174.7	212
213	189.2	95.1	98.3	96.8	90.7	138.0	145.3	166.8	175.5	213
214	190.1	95.6	98.8	97.3	91.2	138.7	146.0	167.5	176.4	214
215	191.0	96.1	99.3	97.8	91.7	139.4	146.7	168.3	177.2	215
216	191.9	96.5	99.8	98.3	92.2	140.0	147.4	169.1	178.0	216
217	192.8	97.0	100.3	98.8	92.7	140.7	148.1	169.9	178.9	217
218	193.6	97.5	100.8	99.3	93.2	141.4	148.8	170.7	179.7	218
219	194.5	98.0	101.2	99.8	93.7	142.0	149.5	171.5	180.5	219
220	195.4	98.4	101.7	100.3	94.2	142.7	150.2	172.3	181.4	220
221	196.3	98.9	102.2	100.8	94.7	143.4	150.9	173.1	182.2	221
222	197.2	99.4	102.7	101.2	95.1	140.0	151.6	173.9	183.0	222
223	198.1	99.9	103.2	101.7	95.6	144.7	152.3	174.7	183.9	223
224	199.0	100.3	103.7	102.2	96.1	145.4	153.0	175.5	184.7	224

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MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (*d*-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	
225	199.9	100.8	104.2	102.7	96.6	146.0	153.7	176.2	185.5	225
226	200.7	101.3	104.6	103.2	97.1	146.7	154.4	177.0	186.4	226
227	201.6	101.8	105.1	103.7	97.6	147.4	155.1	177.8	187.2	227
228	202.5	102.2	105.6	104.2	98.1	148.0	155.8	178.6	188.0	228
229	203.4	102.7	106.1	104.7	98.6	148.7	156.5	179.4	188.8	229
230	204.3	103.2	106.6	105.2	99.1	149.4	157.2	180.2	189.7	230
231	205.2	103.7	107.1	105.7	99.6	150.0	157.9	181.0	190.5	231
232	206.1	104.1	107.6	106.2	100.1	150.7	158.6	181.8	191.3	232
233	207.0	104.6	108.1	196.7	100.6	151.4	159.3	182.6	192.2	233
234	207.9	105.1	108.6	107.2	101.1	152.0	160.0	183.4	193.0	234
235	208.7	105.6	109.1	107.7	101.6	152.7	160.7	184.2	193.8	235
236	209.6	106.0	109.5	108.2	102.1	153.4	161.4	184.9	194.7	236
237	210.5	106.5	110.0	108.7	102.6	154.0	162.1	185.7	195.5	237
238	211.4	107.0	110.5	109.2	103.1	154.7	162.8	186.5	196.3	238
239	212.3	107.5	111.0	109.6	103.5	155.4	163.5	187.3	197.2	239
240	213.2	108.0	111.5	110.1	104.0	156.1	164.3	188.1	198.0	240
241	214.1	108.4	112.0	110.6	104.5	156.7	165.0	188.9	198.8	241
242	215.0	108.9	112.5	111.1	105.0	157.4	165.7	189.7	199.7	242
243	215.8	109.4	113.0	111.6	105.5	158.1	166.4	190.5	200.5	243
244	216.7	109.9	113.5	112.1	106.0	158.7	167.1	191.3	201.3	244
245	217.6	110.4	114.0	112.6	106.5	159.4	167.8	192.1	202.2	245
246	218.5	110.8	114.5	113.1	107.0	160.1	168.5	192.9	203.0	246
247	219.4	111.3	115.0	113.6	107.5	160.7	169.2	193.6	203.8	247
248	220.3	111.8	115.4	114.1	108.0	161.4	169.9	194.4	204.7	248
249	221.2	112.3	115.9	114.6	108.5	162.1	170.6	195.2	205.5	249
250	222.1	112.8	116.4	115.1	109.0	162.7	171.3	196.0	206.3	250
251	223.0	113.2	116.9	115.6	109.5	163.4	172.0	196.8	207.2	251
252	223.8	113.7	117.4	116.1	110.0	164.1	172.7	197.6	208.0	252
253	224.7	114.2	117.9	116.6	110.5	164.7	173.4	198.4	208.8	253
254	225.6	114.7	118.4	117.1	111.0	165.4	174.1	199.2	209.7	254
255	226.5	115.2	118.9	117.6	111.5	166.1	174.8	200.0	210.5	255
256	227.4	115.7	119.4	118.1	112.0	166.8	175.5	200.8	211.3	256
257	228.3	116.1	119.9	118.6	112.5	167.4	176.2	201.6	212.2	257
258	229.2	116.6	120.4	119.1	113.0	168.1	176.9	202.3	213.0	258
259	230.1	117.1	120.9	119.6	113.5	168.8	177.6	203.1	213.8	259
260	231.0	117.6	121.4	120.1	114.0	169.4	178.3	203.9	214.7	260
261	231.8	118.1	121.9	120.6	114.5	170.1	179.0	204.7	215.5	261
262	232.7	118.6	122.4	121.1	115.0	170.8	179.8	205.5	216.3	262
263	233.6	119.0	122.9	121.6	115.5	171.4	180.5	206.3	217.2	263
264	234.5	119.5	123.4	122.1	116.0	172.1	181.2	207.1	218.0	264
265	235.4	120.0	123.9	122.6	116.5	172.8	181.9	207.9	218.8	265
266	236.3	120.5	124.4	123.1	117.0	173.5	182.6	208.7	219.7	266
267	237.2	121.0	124.9	123.6	117.5	174.1	183.3	209.5	220.5	267
268	238.1	121.5	125.4	124.1	118.0	174.8	184.0	210.3	221.3	268
269	238.9	122.0	125.9	124.6	118.5	175.5	184.7	211.0	222.1	269

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Munson and Walker's Table.—Continued.

[Expressed in milligrams.]

Cuprous oxid (Cu_2O)	Copper (Cu)	Dextrose (*d*-glucose)	Invert sugar	Invert sugar and sucrose		Lactose		Maltose		Cuprous oxid (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	
270	239.8	122.5	126.4	125.1	119.0	176.1	185.4	211.8	223.0	270
271	240.7	122.9	126.9	125.6	119.5	176.8	186.1	212.6	223.8	271
272	241.6	123.4	127.4	126.2	120.0	177.5	186.8	213.4	224.6	272
273	242.5	123.9	127.9	126.7	120.6	178.1	187.5	214.2	225.5	273
274	243.4	124.4	128.4	127.2	121.1	178.8	188.2	215.0	226.3	274
275	244.3	124.9	128.9	127.7	121.6	179.5	188.9	215.8	227.1	275
276	245.2	125.4	129.4	128.2	122.1	180.2	189.6	216.6	228.0	276
277	246.1	125.9	129.9	128.7	122.6	180.8	190.3	217.4	228.8	277
278	246.9	126.4	130.4	129.2	123.1	181.5	191.0	218.2	229.6	278
279	247.8	126.9	130.9	129.7	123.6	182.2	191.7	218.9	230.5	279
280	248.7	127.3	131.4	130.2	124.1	182.8	192.4	219.7	231.3	280
281	249.6	127.8	131.9	130.7	124.6	183.5	193.1	220.5	232.1	281
282	250.5	128.3	132.4	131.2	125.1	184.2	193.9	221.3	233.0	282
283	251.4	128.8	132.9	131.7	125.6	184.8	194.6	222.1	233.8	283
284	252.3	129.3	133.4	132.2	126.1	185.5	195.3	222.9	234.6	284
285	253.2	129.8	133.9	132.7	126.6	186.2	196.0	223.7	235.5	285
286	254.0	130.3	134.4	133.2	127.1	186.9	196.7	224.5	236.3	286
287	254.9	130.8	134.9	133.7	127.6	187.5	197.4	225.3	237.1	287
288	255.8	131.3	135.4	134.3	128.1	188.2	198.1	226.1	238.0	288
289	256.7	131.8	135.9	134.8	128.6	188.9	198.8	226.9	238.8	289
290	257.6	132.3	136.4	135.3	129.2	189.5	199.5	227.6	239.6	290
291	258.5	132.7	136.9	135.8	129.7	190.2	200.2	228.4	240.5	291
292	259.4	133.2	137.4	136.3	130.2	190.9	200.9	229.2	241.3	292
293	260.3	133.7	137.9	136.8	130.7	191.5	201.6	230.0	242.1	293
294	261.2	134.2	138.4	137.3	131.2	192.2	202.3	230.8	242.9	294
295	262.0	134.7	138.9	137.8	131.7	192.9	203.0	231.6	243.8	295
296	262.9	135.2	139.4	138.3	132.2	193.6	203.7	232.4	244.6	296
297	263.8	135.7	140.0	138.8	132.7	194.2	204.4	233.2	245.4	297
298	264.7	136.2	140.5	139.4	133.2	194.9	205.1	234.0	246.3	298
299	265.6	136.7	141.0	139.9	133.7	195.6	205.8	234.8	247.1	299
300	266.5	137.2	141.5	140.4	134.2	196.2	206.6	235.5	247.9	300
301	267.4	137.7	142.0	140.9	134.8	196.9	207.3	236.3	248.8	301
302	268.3	138.2	142.5	141.4	135.3	197.6	208.0	237.1	249.6	302
303	269.1	138.7	143.0	141.9	135.8	198.3	208.7	237.9	250.4	303
304	270.0	139.2	143.5	142.4	136.3	198.9	209.4	238.7	251.3	304
305	270.9	139.7	144.0	142.9	136.8	199.6	210.1	239.5	252.1	305
306	271.8	140.2	144.5	143.4	137.3	200.3	210.8	240.3	252.9	306
307	272.7	140.7	145.0	144.0	137.8	201.0	211.5	241.1	253.8	307
308	273.6	141.2	145.5	144.5	138.3	201.6	212.2	241.9	254.6	308
309	274.5	141.7	146.1	145.0	138.8	202.3	212.9	242.7	255.4	309
310	275.4	142.2	146.6	145.5	139.4	203.0	213.7	243.5	256.3	310
311	276.3	142.7	147.1	146.0	139.9	203.6	214.4	244.2	257.1	311
312	277.1	143.2	147.6	146.5	140.4	204.3	215.1	245.0	257.9	312
313	278.0	143.7	148.1	147.0	140.9	205.0	215.8	245.8	258.8	313
314	278.9	144.2	148.6	147.6	141.4	205.7	216.5	246.6	259.6	314

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MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (*d*-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	
315	279.8	144.7	149.1	148.1	141.9	206.3	217.2	247.4	260.4	315
316	280.7	145.2	149.6	148.6	142.4	207.0	217.9	248.2	261.2	316
317	281.6	145.7	150.1	149.1	143.0	207.7	218.6	249.0	262.1	317
318	282.5	146.2	150.7	149.6	143.5	208.4	219.3	249.8	262.9	318
319	283.4	146.7	151.2	150.1	144.0	209.0	220.0	250.6	263.7	319
320	284.2	147.2	151.7	150.7	144.5	209.7	220.7	251.3	264.6	320
321	285.1	147.7	152.2	151.2	145.0	210.4	221.4	252.1	265.4	321
322	286.0	148.2	152.7	151.7	145.5	211.0	222.2	252.9	266.2	322
323	286.9	148.7	153.2	152.2	146.0	211.7	222.9	253.7	267.1	323
324	287.8	149.2	153.7	152.7	146.6	212.4	223.6	254.5	267.9	324
325	288.7	149.7	154.3	153.2	147.1	213.1	224.3	255.3	268.7	325
326	289.6	150.2	154.8	153.8	147.6	213.7	225.0	256.1	269.6	326
327	290.5	150.7	155.3	154.3	148.1	214.4	225.7	256.9	270.4	327
328	291.4	151.2	155.8	154.8	148.6	215.1	226.4	257.7	271.2	328
329	292.2	151.7	156.3	155.3	149.1	215.8	227.1	258.5	272.1	329
330	293.1	152.2	156.8	155.8	149.7	216.4	227.8	259.3	272.9	330
331	294.0	152.7	157.3	156.4	150.2	217.1	228.5	260.0	273.7	331
332	294.9	153.2	157.9	156.9	150.7	217.8	229.2	260.8	274.6	332
333	295.8	153.7	158.4	157.4	151.2	218.4	230.0	261.6	275.4	333
334	296.7	154.2	158.9	157.9	151.7	219.1	230.7	262.4	276.2	334
335	297.6	154.7	159.4	158.4	152.3	219.8	231.4	263.2	277.0	335
336	298.5	155.2	159.9	159.0	152.8	220.5	232.1	254.0	277.9	336
337	299.3	155.8	160.5	159.5	153.3	221.1	232.8	264.8	278.7	337
338	300.2	156.3	161.0	160.0	153.8	221.8	233.5	265.6	279.5	338
339	301.1	156.8	161.5	160.5	154.3	222.5	234.2	266.4	280.4	339
340	302.0	157.3	162.0	161.0	154.8	223.2	234.9	267.1	281.2	340
341	302.9	157.8	162.5	161.6	155.4	223.8	235.6	267.9	282.0	341
342	303.8	158.3	163.1	162.1	155.9	224.5	236.3	268.7	282.9	342
343	304.7	158.8	163.6	162.6	156.4	225.2	237.0	269.5	283.7	343
344	305.6	159.3	164.1	163.1	156.9	225.9	237.8	270.3	284.5	344
345	306.5	159.8	164.6	163.7	157.5	226.5	238.5	271.1	285.4	345
346	307.3	160.3	165.1	164.2	158.0	227.2	239.2	271.9	286.2	346
347	308.2	160.8	165.7	164.7	158.5	227.9	239.9	272.7	287.0	347
348	309.1	161.4	166.2	165.2	159.0	228.5	240.6	273.5	287.9	348
349	310.0	161.9	166.7	165.7	159.5	229.2	241.3	274.3	288.7	349
350	310.9	162.4	167.2	166.3	160.1	229.9	242.0	275.0	289.5	350
351	311.8	162.9	167.7	166.8	160.6	230.6	242.7	275.8	290.4	351
352	312.7	163.4	168.3	167.3	161.1	231.2	243.4	276.6	291.2	352
353	313.6	163.9	168.8	167.8	161.6	231.9	244.1	277.4	292.0	353
354	314.4	164.4	169.3	168.4	162.2	232.6	244.8	278.2	292.8	354
355	315.3	164.9	169.8	168.9	162.7	233.3	245.6	279.0	293.7	355
356	316.2	165.4	170.4	169.4	163.2	233.9	246.3	279.8	294.5	356
357	317.1	166.0	170.9	170.0	163.7	234.6	247.0	280.6	295.3	357
358	318.0	166.5	171.4	170.5	164.3	235.3	247.7	281.4	296.2	358
359	318.9	167.0	171.9	171.0	164.8	236.0	248.4	282.2	297.0	359

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MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (d-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	
360	319.8	167.5	172.5	171.5	165.3	236.7	249.1	282.9	297.8	360
361	320.7	168.0	173.0	172.1	165.8	237.3	249.8	283.7	298.7	361
362	321.6	168.5	173.5	172.6	166.4	238.0	250.5	284.5	299.5	362
363	322.4	169.0	174.0	173.1	166.9	238.7	251.2	285.3	300.3	363
364	323.3	169.6	174.6	173.7	167.4	239.4	252.0	286.1	301.2	364
365	324.2	170.1	175.1	174.2	167.9	240.0	252.7	286.9	302.0	365
366	325.1	170.6	175.6	174.7	168.5	240.7	253.4	287.7	302.8	366
367	326.0	171.1	176.1	175.2	169.0	241.4	254.1	288.5	303.6	367
368	326.9	171.6	176.7	175.8	169.5	242.1	254.8	289.3	304.5	368
369	327.8	172.1	177.2	176.3	170.0	242.7	255.5	290.0	305.3	369
370	328.7	172.7	177.7	176.8	170.6	243.4	256.2	290.8	306.1	370
371	329.5	173.2	178.3	177.4	171.1	244.1	256.9	291.6	307.0	371
372	330.4	173.7	178.8	177.9	171.6	244.8	257.7	292.4	307.8	372
373	331.3	174.2	179.3	178.4	172.2	245.4	258.4	293.2	308.6	373
374	332.2	174.7	179.8	179.0	172.7	246.1	259.1	294.0	309.5	374
375	333.1	175.3	180.4	179.5	173.2	246.8	259.8	294.8	310.3	375
376	334.0	175.8	180.9	180.0	173.7	247.5	260.5	295.6	311.1	376
377	334.9	176.3	181.4	180.6	174.3	248.1	261.2	296.4	312.0	377
378	335.8	176.8	182.0	181.1	174.8	248.8	261.9	297.2	312.8	378
379	336.7	177.3	182.5	181.6	175.3	249.5	262.6	297.9	313.6	379
380	337.5	177.9	183.0	182.1	175.9	250.2	263.4	298.7	314.5	380
381	338.4	178.4	183.6	182.7	176.4	250.8	264.1	299.5	315.3	381
382	339.3	178.9	184.1	183.2	176.9	251.5	264.8	300.3	316.1	382
383	340.2	179.4	184.6	183.8	177.5	252.2	265.5	301.1	316.9	383
384	341.1	180.0	185.2	184.3	178.0	252.9	266.2	301.9	317.8	384
385	342.0	180.5	185.7	184.8	178.5	253.6	266.9	302.7	318.6	385
386	342.9	181.0	186.2	185.4	179.1	254.2	267.6	303.5	319.4	386
387	343.8	181.5	186.8	185.9	179.6	254.9	268.3	304.2	320.3	387
388	344.6	182.0	187.3	186.4	180.1	255.6	269.0	305.0	321.1	388
389	345.5	182.6	187.8	187.0	180.6	256.3	269.8	305.8	321.9	389
390	346.4	183.1	188.4	187.5	181.2	256.9	270.5	306.6	322.8	390
391	347.3	183.6	188.9	188.0	181.7	257.6	271.2	307.4	323.6	391
392	348.2	184.1	189.4	188.6	182.3	258.3	271.9	308.2	324.4	392
393	349.1	184.7	190.0	189.1	182.8	259.0	272.6	309.0	325.2	393
394	350.0	185.2	190.5	189.7	183.3	259.6	273.3	309.8	326.1	394
395	350.9	185.7	191.0	190.2	183.9	260.3	274.0	310.6	326.9	395
396	351.8	186.2	191.6	190.7	184.4	261.0	274.7	311.4	327.7	396
397	352.6	186.8	192.1	191.3	184.9	261.7	275.5	312.1	328.6	397
398	353.5	187.3	192.7	191.8	185.5	262.3	276.2	312.9	329.4	398
399	354.4	187.8	193.2	192.3	186.0	263.0	276.9	313.7	330.2	399
400	355.3	188.4	193.7	192.9	186.5	263.7	277.6	314.5	331.1	400
401	356.2	188.9	194.3	193.4	187.1	264.4	278.3	315.3	331.9	401
402	357.1	189.4	194.8	194.0	187.6	265.0	279.0	316.1	332.7	402
403	358.0	189.9	195.4	194.5	188.1	265.7	279.7	316.9	333.6	403
404	358.9	190.5	195.9	195.0	188.7	266.4	280.4	317.7	334.4	404

1

MUNSON AND WALKER'S TABLE.—Continued.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (*d*-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}.H_2O$	
405	359.7	191.0	196.4	195.6	189.2	267.1	281.1	318.5	335.2	405
406	360.6	191.5	197.0	196.1	189.8	267.8	281.9	319.2	336.0	406
407	361.5	192.1	197.5	196.7	190.3	268.4	282.6	320.0	336.9	407
408	362.4	192.6	198.1	197.2	190.8	269.1	283.3	320.8	337.7	408
409	363.3	193.1	198.6	197.7	191.4	269.8	284.0	321.6	338.5	409
410	364.2	193.7	199.1	198.3	191.9	270.5	284.7	322.4	339.4	410
411	365.1	194.2	199.7	198.8	192.5	271.2	285.4	323.2	340.2	411
412	366.0	194.7	200.2	199.4	193.0	271.8	286.2	324.0	341.0	412
413	366.9	195.2	200.8	199.9	193.5	272.5	286.9	324.8	341.9	413
414	367.7	195.8	201.3	200.5	194.1	273.2	287.6	325.6	342.7	414
415	368.6	196.3	201.8	201.0	194.6	273.9	288.3	326.3	343.5	415
416	369.5	196.8	202.4	201.6	195.2	274.6	289.0	327.1	344.4	416
417	370.4	197.4	202.9	202.1	195.7	275.2	289.7	327.9	345.2	417
418	371.3	197.9	203.5	202.6	196.2	275.9	290.4	328.7	346.0	418
419	372.2	198.4	204.0	203.2	196.8	276.6	291.2	329.5	346.8	419
420	373.1	199.0	204.6	203.7	197.3	277.3	291.9	330.3	347.7	420
421	374.0	199.5	205.1	204.3	197.9	277.9	292.6	331.1	348.5	421
422	374.8	200.1	205.7	204.8	198.4	278.6	293.3	331.9	349.3	422
423	375.7	200.6	206.2	205.4	198.9	279.3	294.0	332.7	350.2	423
424	376.6	201.1	206.7	205.9	199.5	280.0	294.7	333.4	351.0	424
425	377.5	201.7	207.3	206.5	200.0	280.7	295.4	334.2	351.8	425
426	378.4	202.2	207.8	207.0	200.6	281.3	296.2	335.0	352.7	426
427	379.3	202.8	208.4	207.6	201.1	282.0	296.9	335.8	353.5	427
428	380.2	203.3	208.9	208.1	201.7	282.7	297.6	336.6	354.3	428
429	381.1	203.8	209.5	208.7	202.2	283.4	298.3	337.4	355.1	429
430	382.0	204.4	210.0	209.2	202.7	284.1	299.0	338.2	356.0	430
431	382.8	204.9	210.6	209.8	203.3	284.7	299.7	339.0	356.8	431
432	383.7	205.5	211.1	210.3	203.8	285.4	300.5	339.7	357.6	432
433	384.6	206.0	211.7	210.9	204.4	286.1	301.2	340.5	358.5	433
434	385.5	206.5	212.2	211.4	204.9	286.8	301.9	341.3	359.3	434
435	386.4	207.1	212.8	212.0	205.5	287.5	302.6	342.1	360.1	435
436	387.3	207.6	213.3	212.5	206.0	288.1	303.3	342.9	361.0	436
437	388.2	208.2	213.9	213.1	206.6	288.8	304.0	343.7	361.8	437
438	389.1	208.7	214.4	213.6	207.1	289.5	304.7	344.5	362.6	438
439	390.0	209.2	215.0	214.2	207.7	290.2	305.5	345.3	363.4	439
440	390.8	209.8	215.5	214.7	208.2	290.9	306.2	346.1	364.3	440
441	391.7	210.3	216.1	215.3	208.8	291.5	306.9	346.8	365.1	441
442	392.6	210.9	216.6	215.8	209.3	292.2	307.6	347.6	365.9	442
443	393.5	211.4	217.2	216.4	209.9	292.9	308.3	348.4	366.8	443
444	394.4	212.0	217.8	216.9	210.4	293.6	309.0	349.2	367.6	444
445	395.3	212.5	218.3	217.5	211.0	294.2	309.7	350.0	368.4	445
446	396.2	213.1	218.9	218.0	211.5	294.9	310.5	350.8	369.3	446
447	397.1	213.6	219.4	218.6	212.1	295.6	311.2	351.6	370.1	447
448	397.9	214.1	220.0	219.1	212.6	296.3	311.9	352.4	370.9	448
449	398.8	214.7	220.5	219.7	213.2	297.0	312.6	353.2	371.7	449

1

MUNSON AND WALKER'S TABLE.—Concluded.

[Expressed in milligrams.]

CUPROUS OXID (Cu_2O)	COPPER (Cu)	DEXTROSE (*d*-GLUCOSE)	INVERT SUGAR	INVERT SUGAR AND SUCROSE		LACTOSE		MALTOSE		CUPROUS OXID (Cu_2O)
				0.4 gram total sugar	2 grams total sugar	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	$C_{12}H_{22}O_{11}$	$C_{12}H_{22}O_{11}H_2O$	
450	399.7	215.2	221.1	220.2	213.7	297.6	313.3	353.9	372.6	450
451	400.6	215.8	221.6	220.8	214.3	298.3	314.0	354.7	373.4	451
452	401.5	216.3	222.2	221.4	214.8	299.0	314.7	355.5	374.2	452
453	402.4	216.9	222.8	221.9	215.4	299.7	315.5	356.3	375.1	453
454	403.3	217.4	223.3	222.5	215.9	300.4	316.2	357.1	375.9	454
455	404.2	218.0	223.9	223.0	216.5	301.1	316.9	357.9	376.7	455
456	405.1	218.5	224.4	223.6	217.0	301.7	317.6	358.7	377.6	456
457	405.9	219.1	225.0	224.1	217.6	302.4	318.3	359.5	378.4	457
458	406.8	219.6	225.5	224.7	218.1	303.1	319.0	360.3	379.2	458
459	407.7	220.2	226.7	225.3	218.7	303.8	319.8	361.0	380.0	459
460	408.6	220.7	226.7	225.8	219.2	304.5	320.5	361.8	380.9	460
461	409.5	221.3	227.2	226.4	219.8	305.1	321.2	362.6	381.7	461
462	410.4	221.8	227.8	226.9	220.3	305.8	321.9	363.4	382.5	462
463	411.3	222.4	228.3	227.5	220.9	306.5	322.6	364.2	383.4	463
464	412.2	222.9	228.9	228.1	221.4	307.2	323.4	365.0	384.2	464
465	413.0	223.5	229.5	228.6	222.0	307.9	324.1	365.8	385.0	465
466	413.9	224.0	230.0	229.2	222.5	308.6	324.8	366.6	385.9	466
467	414.8	224.6	230.6	229.7	223.1	309.2	325.5	367.3	386.7	467
468	415.7	225.1	231.2	230.3	223.7	309.9	326.2	368.1	387.5	468
469	416.6	225.7	231.7	230.9	224.2	310.6	326.9	368.9	388.3	469
470	417.5	226.2	232.3	231.4	224.8	311.3	327.7	369.7	389.2	470
471	418.4	226.8	232.8	232.0	225.3	312.0	328.4	370.5	390.0	471
472	419.3	227.4	233.4	233.4	225.9	312.6	329.1	371.3	390.8	472
473	420.2	227.9	234.0	233.1	226.4	313.3	329.8	372.1	391.7	473
474	421.0	228.5	234.5	233.7	227.0	314.0	330.5	372.9	392.5	474
475	421.9	229.0	235.1	234.2	227.6	314.7	331.3	373.7	393.3	475
476	422.8	229.6	235.7	234.8	228.1	315.4	332.0	374.4	394.2	476
477	423.7	230.1	236.2	235.4	228.7	316.1	332.7	375.2	395.0	477
478	424.6	230.7	236.8	235.9	229.2	316.7	333.4	376.0	395.8	478
479	425.5	231.3	237.4	236.5	229.8	317.4	334.1	376.8	396.6	479
480	426.4	231.8	237.9	237.1	230.3	318.1	334.8	377.6	397.5	480
481	427.3	232.4	238.5	237.6	230.9	318.8	335.6	378.4	398.3	481
482	428.1	232.9	239.1	238.2	231.5	319.5	336.3	379.2	399.1	482
483	429.0	233.5	239.6	238.8	232.0	320.1	337.0	380.0	400.0	483
484	429.9	234.1	240.2	239.3	232.6	320.8	337.7	380.7	400.8	484
485	430.8	234.6	240.8	239.9	233.2	321.5	338.4	381.5	401.6	485
486	431.7	235.2	241.4	240.5	233.7	322.2	339.1	382.3	402.4	486
487	432.6	235.7	241.9	241.0	234.3	322.9	339.9	383.1	403.3	487
488	433.5	236.3	242.5	241.6	234.8	323.6	340.6	383.9	404.1	488
489	434.4	236.9	243.1	242.2	235.4	324.2	341.3	384.7	404.9	489
490	435.3	237.4	243.6	242.7	236.0	324.9	342.0	385.5	405.8	490

2

KRÖBER'S TABLE.

For Determining Pentoses and Pentosans.

[Expressed in grams.]

FURFURAL PHLOROGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.030	0.0182	0.0391	0.0344	0.0324	0.0285	0.0358	0.0315
0.031	0.0188	0.0402	0.0354	0.0333	0.0293	0.0368	0.0324
0.032	0.0193	0.0413	0.0363	0.0342	0.0301	0.0378	0.0333
0.033	0.0198	0.0424	0.0373	0.0352	0.0309	0.0388	0.0341
0.034	0.0203	0.0435	0.0383	0.0361	0.0317	0.0398	0.0350
0.035	0.0209	0.0446	0.0393	0.0370	0.0326	0.0408	0.0359
0.036	0.0214	0.0457	0.0402	0.0379	0.0334	0.0418	0.0368
0.037	0.0219	0.0468	0.0412	0.0388	0.0342	0.0428	0.0377
0.038	0.0224	0.0479	0.0422	0.0398	0.0350	0.0439	0.0386
0.039	0.0229	0.0490	0.0431	0.0407	0.0358	0.0449	0.0395
0.040	0.0235	0.0501	0.0441	0.0416	0.0366	0.0459	0.0404
0.041	0.0240	0.0512	0.0451	0.0425	0.0374	0.0469	0.0413
0.042	0.0245	0.0523	0.0460	0.0434	0.0382	0.0479	0.0422
0.043	0.0250	0.0534	0.0470	0.0443	0.0390	0.0489	0.0431
0.044	0.0255	0.0545	0.0480	0.0452	0.0398	0.0499	0.0440
0.045	0.0260	0.0556	0.0490	0.0462	0.0406	0.0509	0.0448
0.046	0.0266	0.0567	0.0499	0.0471	0.0414	0.0519	0.0457
0.047	0.0271	0.0578	0.0509	0.0480	0.0422	0.0529	0.0466
0.048	0.0276	0.0589	0.0519	0.0489	0.0430	0.0539	0.0475
0.049	0.0281	0.0600	0.0528	0.0498	0.0438	0.0549	0.0484
0.050	0.0286	0.0611	0.0538	0.0507	0.0446	0.0559	0.0492
0.051	0.0292	0.0622	0.0548	0.0516	0.0454	0.0569	0.0501
0.052	0.0297	0.0633	0.0557	0.0525	0.0462	0.0579	0.0510
0.053	0.0302	0.0644	0.0567	0.0534	0.0470	0.0589	0.0519
0.054	0.0307	0.0655	0.0576	0.0543	0.0478	0.0599	0.0528
0.055	0.0312	0.0666	0.0586	0.0553	0.0486	0.0610	0.0537
0.056	0.0318	0.0677	0.0596	0.0562	0.0494	0.0620	0.0546
0.057	0.0323	0.0688	0.0605	0.0571	0.0502	0.0630	0.0555
0.058	0.0328	0.0699	0.0615	0.0580	0.0510	0.0640	0.0564
0.059	0.0333	0.0710	0.0624	0.0589	0.0518	0.0650	0.0573
0.060	0.0338	0.0721	0.0634	0.0598	0.0526	0.0660	0.0581
0.061	0.0344	0.0732	0.0644	0.0607	0.0534	0.0670	0.0590
0.062	0.0349	0.0743	0.0653	0.0616	0.0542	0.0680	0.0599
0.063	0.0354	0.0754	0.0663	0.0626	0.0550	0.0690	0.0608
0.064	0.0359	0.0765	0.0673	0.0635	0.0558	0.0700	0.0617
0.065	0.0364	0.0776	0.0683	0.0644	0.0567	0.0710	0.0625
0.066	0.0370	0.0787	0.0692	0.0653	0.0575	0.0720	0.0634
0.067	0.0375	0.0798	0.0702	0.0662	0.0583	0.0730	0.0643
0.068	0.0380	0.0809	0.0712	0.0672	0.0591	0.0741	0.0652
0.069	0.0385	0.0820	0.0721	0.0681	0.0599	0.0751	0.0661
0.070	0.0390	0.0831	0.0731	0.0690	0.0607	0.0761	0.0670
0.071	0.0396	0.0842	0.0741	0.0699	0.0615	0.0771	0.0679
0.072	0.0401	0.0853	0.0750	0.0708	0.0623	0.0781	0.0688
0.073	0.0406	0.0864	0.0760	0.0717	0.0631	0.0791	0.0697
0.074	0.0411	0.0875	0.0770	0.0726	0.0639	0.0801	0.0706

2

KRÖBER'S TABLE.—Continued.

[Expressed in grams.]

FURFURAL PHLOROGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.075	0.0416	0.0886	0.0780	0.0736	0.0647	0.0811	0.0714
0.076	0.0422	0.0897	0.0789	0.0745	0.0655	0.0821	0.0722
0.077	0.0427	0.0908	0.0799	0.0754	0.0663	0.0831	0.0731
0.078	0.0432	0.0919	0.0809	0.0763	0.0671	0.0841	0.0740
0.079	0.0437	0.0930	0.0818	0.0772	0.0679	0.0851	0.0749
0.080	0.0442	0.0941	0.0828	0.0781	0.0687	0.0861	0.0758
0.081	0.0448	0.0952	0.0838	0.0790	0.0695	0.0871	0.0767
0.082	0.0453	0.0963	0.0847	0.0799	0.0703	0.0881	0.0776
0.083	0.0458	0.0974	0.0857	0.0808	0.0711	0.0891	0.0785
0.084	0.0463	0.0985	0.0867	0.0817	0.0719	0.0901	0.0794
0.085	0.0468	0.0996	0.0877	0.0827	0.0727	0.0912	0.0803
0.086	0.0474	0.1007	0.0886	0.0836	0.0735	0.0922	0.0812
0.087	0.0479	0.1018	0.0896	0.0845	0.0743	0.0932	0.0821
0.088	0.0484	0.1029	0.0906	0.0854	0.0751	0.0942	0.0830
0.089	0.0489	0.1040	0.0915	0.0863	0.0759	0.0952	0.0838
0.090	0.0494	0.1051	0.0925	0.0872	0.0767	0.0962	0.0847
0.091	0.0499	0.1062	0.0935	0.0881	0.0775	0.0972	0.0856
0.092	0.0505	0.1073	0.0944	0.0890	0.0783	0.0982	0.0865
0.093	0.0510	0.1084	0.0954	0.0900	0.0791	0.0992	0.0874
0.094	0.0515	0.1095	0.0964	0.0909	0.0800	0.1002	0.0883
0.095	0.0520	0.1106	0.0974	0.0918	0.0808	0.1012	0.0891
0.096	0.0525	0.1117	0.0983	0.0927	0.0816	0.1022	0.0899
0.097	0.0531	0.1128	0.0993	0.0936	0.0824	0.1032	0.0908
0.098	0.0536	0.1139	0.1003	0.0946	0.0832	0.1043	0.0917
0.099	0.0541	0.1150	0.1012	0.0955	0.0840	0.1053	0.0926
0.100	0.0546	0.1161	0.1022	0.0964	0.0848	0.1063	0.0935
0.101	0.0551	0.1171	0.1032	0.0973	0.0856	0.1073	0.0944
0.102	0.0557	0.1182	0.1041	0.0982	0.0864	0.1083	0.0953
0.103	0.0562	0.1193	0.1051	0.0991	0.0872	0.1093	0.0962
0.104	0.0567	0.1204	0.1060	0.1000	0.0880	0.1103	0.0971
0.105	0.0572	0.1215	0.1070	0.1010	0.0888	0.1113	0.0979
0.106	0.0577	0.1226	0.1080	0.1019	0.0896	0.1123	0.0988
0.107	0.0582	0.1237	0.1089	0.1028	0.0904	0.1133	0.0997
0.108	0.0588	0.1248	0.1099	0.1037	0.0912	0.1143	0.1006
0.109	0.0593	0.1259	0.1108	0.1046	0.0920	0.1153	0.1015
0.110	0.0598	0.1270	0.1118	0.1055	0.0928	0.1163	0.1023
0.111	0.0603	0.1281	0.1128	0.1064	0.0936	0.1173	0.1032
0.112	0.0608	0.1292	0.1137	0.1073	0.0944	0.1183	0.1041
0.113	0.0614	0.1303	0.1147	0.1082	0.0952	0.1193	0.1050
0.114	0.0619	0.1314	0.1156	0.1091	0.0960	0.1203	0.1059
0.115	0.0624	0.1325	0.1166	0.1101	0.0968	0.1213	0.1067
0.116	0.0629	0.1336	0.1176	0.1110	0.0976	0.1223	0.1076
0.117	0.0634	0.1347	0.1185	0.1119	0.0984	0.1233	0.1085
0.118	0.0640	0.1358	0.1195	0.1128	0.0992	0.1243	0.1094
0.119	0.0645	0.1369	0.1204	0.1137	0.1000	0.1253	0.1103

2

Kröber's Table.—Continued.

[Expressed in grams.]

FURFURAL PHLOROGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.120	0.0650	0.1380	0.1214	0.1146	0.1008	0.1263	0.1111
0.121	0.0655	0.1391	0.1224	0.1155	0.1016	0.1273	0.1120
0.122	0.0660	0.1402	0.1233	0.1164	0.1024	0.1283	0.1129
0.123	0.0665	0.1413	0.1243	0.1173	0.1032	0.1293	0.1138
0.124	0.0671	0.1424	0.1253	0.1182	0.1040	0.1303	0.1147
0.125	0.0676	0.1435	0.1263	0.1192	0.1049	0.1314	0.1156
0.126	0.0681	0.1446	0.1272	0.1201	0.1057	0.1324	0.1165
0.127	0.0686	0.1457	0.1282	0.1210	0.1065	0.1334	0.1174
0.128	0.0691	0.1468	0.1292	0.1219	0.1073	0.1344	0.1183
0.129	0.0697	0.1479	0.1301	0.1228	0.1081	0.1354	0.1192
0.130	0.0702	0.1490	0.1311	0.1237	0.1089	0.1364	0.1201
0.131	0.0707	0.1501	0.1321	0.1246	0.1097	0.1374	0.1210
0.132	0.0712	0.1512	0.1330	0.1255	0.1105	0.1384	0.1219
0.133	0.0717	0.1523	0.1340	0.1264	0.1113	0.1394	0.1227
0.134	0.0723	0.1534	0.1350	0.1273	0.1121	0.1404	0.1236
0.135	0.0728	0.1545	0.1360	0.1283	0.1129	0.1414	0.1244
0.136	0.0733	0.1556	0.1369	0.1292	0.1137	0.1424	0.1253
0.137	0.0738	0.1567	0.1379	0.1301	0.1145	0.1434	0.1262
0.138	0.0743	0.1578	0.1389	0.1310	0.1153	0.1444	0.1271
0.139	0.0748	0.1589	0.1398	0.1319	0.1161	0.1454	0.1280
0.140	0.0754	0.1600	0.1408	0.1328	0.1169	0.1464	0.1288
0.141	0.0759	0.1611	0.1418	0.1337	0.1177	0.1474	0.1297
0.142	0.0764	0.1622	0.1427	0.1346	0.1185	0.1484	0.1306
0.143	0.0769	0.1633	0.1437	0.1355	0.1193	0.1494	0.1315
0.144	0.0774	0.1644	0.1447	0.1364	0.1201	0.1504	0.1324
0.145	0.0780	0.1655	0.1457	0.1374	0.1209	0.1515	0.1333
0.146	0.0785	0.1666	0.1466	0.1383	0.1217	0.1525	0.1342
0.147	0.0790	0.1677	0.1476	0.1392	0.1225	0.1535	0.1351
0.148	0.0795	0.1688	0.1486	0.1401	0.1233	0.1545	0.1360
0.149	0.0800	0.1699	0.1495	0.1410	0.1241	0.1555	0.1369
0.150	0.0805	0.1710	0.1505	0.1419	0.1249	0.1565	0.1377
0.151	0.0811	0.1721	0.1515	0.1428	0.1257	0.1575	0.1386
0.152	0.0816	0.1732	0.1524	0.1437	0.1265	0.1585	0.1395
0.153	0.0821	0.1743	0.1534	0.1446	0.1273	0.1595	0.1404
0.154	0.0826	0.1754	0.1544	0.1455	0.1281	0.1605	0.1413
0.155	0.0831	0.1765	0.1554	0.1465	0.1289	0.1615	0.1421
0.156	0.0837	0.1776	0.1563	0.1474	0.1297	0.1625	0.1430
0.157	0.0842	0.1787	0.1573	0.1483	0.1305	0.1635	0.1439
0.158	0.0847	0.1798	0.1583	0.1492	0.1313	0.1645	0.1448
0.159	0.0852	0.1809	0.1592	0.1501	0.1321	0.1655	0.1457
0.160	0.0857	0.1820	0.1602	0.1510	0.1329	0.1665	0.1465
0.161	0.0863	0.1831	0.1612	0.1519	0.1337	0.1675	0.1474
0.162	0.0868	0.1842	0.1621	0.1528	0.1345	0.1685	0.1483
0.163	0.0873	0.1853	0.1631	0.1537	0.1353	0.1695	0.1492
0.164	0.0878	0.1864	0.1640	0.1546	0.1361	0.1705	0.1501

2

Kröber's Table.—Continued.

[Expressed in grams.]

FURFURAL PHLOROGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.165	0.0883	0.1875	0.1650	0.1556	0.1369	0.1716	0.1510
0.166	0.0888	0.1886	0.1660	0.1565	0.1377	0.1726	0.1519
0.167	0.0894	0.1897	0.1669	0.1574	0.1385	0.1736	0.1528
0.168	0.0899	0.1908	0.1679	0.1583	0.1393	0.1746	0.1537
0.169	0.0904	0.1919	0.1688	0.1592	0.1401	0.1756	0.1546
0.170	0.0909	0.1930	0.1698	0.1601	0.1409	0.1766	0.1554
0.171	0.0914	0.1941	0.1708	0.1610	0.1417	0.1776	0.1563
0.172	0.0920	0.1952	0.1717	0.1619	0.1425	0.1786	0.1572
0.173	0.0925	0.1963	0.1727	0.1628	0.1433	0.1796	0.1581
0.174	0.0930	0.1974	0.1736	0.1637	0.1441	0.1806	0.1590
0.175	0.0935	0.1985	0.1746	0.1647	0.1449	0.1816	0.1598
0.176	0.0940	0.1996	0.1756	0.1656	0.1457	0.1826	0.1607
0.177	0.0946	0.2007	0.1765	0.1665	0.1465	0.1836	0.1616
0.178	0.0951	0.2018	0.1775	0.1674	0.1473	0.1846	0.1625
0.179	0.0956	0.2029	0.1784	0.1683	0.1481	0.1856	0.1634
0.180	0.0961	0.2039	0.1794	0.1692	0.1489	0.1866	0.1642
0.181	0.0966	0.2050	0.1804	0.1701	0.1497	0.1876	0.1651
0.182	0.0971	0.2061	0.1813	0.1710	0.1505	0.1886	0.1660
0.183	0.0977	0.2072	0.1823	0.1719	0.1513	0.1896	0.1669
0.184	0.0982	0.2082	0.1832	0.1728	0.1521	0.1906	0.1678
0.185	0.0987	0.2093	0.1842	0.1738	0.1529	0.1916	0.1686
0.186	0.0992	0.2104	0.1851	0.1747	0.1537	0.1926	0.1695
0.187	0.0997	0.2115	0.1861	0.1756	0.1545	0.1936	0.1704
0.188	0.1003	0.2126	0.1870	0.1765	0.1553	0.1946	0.1712
0.189	0.1008	0.2136	0.1880	0.1774	0.1561	0.1955	0.1721
0.190	0.1013	0.2147	0.1889	0.1783	0.1569	0.1965	0.1729
0.191	0.1018	0.2158	0.1899	0.1792	0.1577	0.1975	0.1738
0.192	0.1023	0.2168	0.1908	0.1801	0.1585	0.1985	0.1747
0.193	0.1028	0.2179	0.1918	0.1810	0.1593	0.1995	0.1756
0.194	0.1034	0.2190	0.1927	0.1819	0.1601	0.2005	0.1764
0.195	0.1039	0.2201	0.1937	0.1829	0.1609	0.2015	0.1773
0.196	0.1044	0.2212	0.1946	0.1838	0.1617	0.2025	0.1782
0.197	0.1049	0.2222	0.1956	0.1847	0.1625	0.2035	0.1791
0.198	0.1054	0.2233	0.1965	0.1856	0.1633	0.2045	0.1800
0.199	0.1059	0.2244	0.1975	0.1865	0.1641	0.2055	0.1808
0.200	0.1065	0.2255	0.1984	0.1874	0.1649	0.2065	0.1817
0.201	0.1070	0.2266	0.1994	0.1883	0.1657	0.2075	0.1826
0.202	0.1075	0.2276	0.2003	0.1892	0.1665	0.2085	0.1835
0.203	0.1080	0.2287	0.2013	0.1901	0.1673	0.2095	0.1844
0.204	0.1085	0.2298	0.2022	0.1910	0.1681	0.2105	0.1853
0.205	0.1090	0.2309	0.2032	0.1920	0.1689	0.2115	0.1861
0.206	0.1096	0.2320	0.2041	0.1929	0.1697	0.2125	0.1869
0.207	0.1101	0.2330	0.2051	0.1938	0.1705	0.2134	0.1878
0.208	0.1106	0.2341	0.2060	0.1947	0.1713	0.2144	0.1887
0.209	0.1111	0.2352	0.2069	0.1956	0.1721	0.2154	0.1896

2

Kröber's Table.—Continued.

[Expressed in grams.]

FURFURAL PHLOROGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.210	0.1116	0.2363	0.2079	0.1965	0.1729	0.2164	0.1904
0.211	0.1121	0.2374	0.2089	0.1975	0.1737	0.2174	0.1913
0.212	0.1127	0.2384	0.2098	0.1984	0.1745	0.2184	0.1922
0.213	0.1132	0.2395	0.2108	0.1993	0.1753	0.2194	0.1931
0.214	0.1137	0.2406	0.2117	0.2002	0.1761	0.2204	0.1940
0.215	0.1142	0.2417	0.2127	0.2011	0.1770	0.2214	0.1948
0.216	0.1147	0.2428	0.2136	0.2020	0.1778	0.2224	0.1957
0.217	0.1152	0.2438	0.2146	0.2029	0.1786	0.2234	0.1966
0.218	0.1158	0.2449	0.2155	0.2038	0.1794	0.2244	0.1974
0.219	0.1163	0.2460	0.2165	0.2047	0.1802	0.2254	0.1983
0.220	0.1168	0.2471	0.2174	0.2057	0.1810	0.2264	0.1992
0.221	0.1173	0.2482	0.2184	0.2066	0.1818	0.2274	0.2001
0.222	0.1178	0.2492	0.2193	0.2075	0.1826	0.2284	0.2010
0.223	0.1183	0.2503	0.2203	0.2084	0.1834	0.2294	0.2019
0.244	0.1189	0.2514	0.2212	0.2093	0.1842	0.2304	0.2028
0.225	0.1194	0.2525	0.2222	0.2102	0.1850	0.2314	0.2037
0.226	0.1199	0.2536	0.2232	0.2111	0.1858	0.2324	0.2046
0.227	0.1204	0.2546	0.2241	0.2121	0.1866	0.2334	0.2054
0.228	0.1209	0.2557	0.2251	0.2130	0.1874	0.2344	0.2063
0.229	0.1214	0.2568	0.2260	0.2139	0.1882	0.2354	0.2072
0.230	0.1220	0.2579	0.2270	0.2148	0.1890	0.2364	0.2081
0.231	0.1225	0.2590	0.2280	0.2157	0.1898	0.2374	0.2089
0.232	0.1230	0.2600	0.2289	0.2166	0.1906	0.2383	0.2097
0.233	0.1235	0.2611	0.2299	0.2175	0.1914	0.2393	0.2106
0.234	0.1240	0.2622	0.2308	0.2184	0.1922	0.2403	0.2115
0.235	0.1245	0.2633	0.2318	0.2193	0.1930	0.2413	0.2124
0.236	0.1251	0.2644	0.2327	0.2202	0.1938	0.2423	0.2132
0.237	0.1256	0.2654	0.2337	0.2211	0.1946	0.2433	0.2141
0.238	0.1261	0.2665	0.2346	0.2220	0.1954	0.2443	0.2150
0.239	0.1266	0.2676	0.2356	0.2229	0.1962	0.2453	0.2159
0.240	0.1271	0.2687	0.2365	0.2239	0.1970	0.2463	0.2168
0.241	0.1276	0.2698	0.2375	0.2248	0.1978	0.2473	0.2176
0.242	0.1281	0.2708	0.2384	0.2257	0.1986	0.2483	0.2185
0.243	0.1287	0.2719	0.2394	0.2266	0.1994	0.2493	0.2194
0.244	0.1292	0.2730	0.2403	0.2275	0.2002	0.2503	0.2203
0.245	0.1297	0.2741	0.2413	0.2284	0.2010	0.2513	0.2212
0.246	0.1302	0.2752	0.2422	0.2293	0.2018	0.2523	0.2220
0.247	0.1307	0.2762	0.2432	0.2302	0.2026	0.2533	0.2229
0.248	0.1312	0.2773	0.2441	0.2311	0.2034	0.2543	0.2238
0.249	0.1318	0.2784	0.2451	0.2320	0.2042	0.2553	0.2247
0.250	0.1323	0.2795	0.2460	0.2330	0.2050	0.2563	0.2256
0.251	0.1328	0.2806	0.2470	0.2339	0.2058	0.2573	0.2264
0.252	0.1333	0.2816	0.2479	0.2348	0.2066	0.2582	0.2272
0.253	0.1338	0.2827	0.2489	0.2357	0.2074	0.2592	0.2281
0.254	0.1343	0.2838	0.2498	0.2366	0.2082	0.2602	0.2290

2

Kröber's Table.—Concluded.

[Expressed in grams.]

FURFURAL PHLOROGLUCID	FURFURAL	ARABINOSE	ARABAN	XYLOSE	XYLAN	PENTOSE	PENTOSAN
0.255	0.1349	0.2849	0.2508	0.2375	0.2090	0.2612	0.2299
0.256	0.1354	0.2860	0.2517	0.2384	0.2098	0.2622	0.2307
0.257	0.1359	0.2870	0.2526	0.2393	0.2106	0.2632	0.2316
0.258	0.1364	0.2881	0.2536	0.2402	0.2114	0.2642	0.2325
0.259	0.1369	0.2892	0.2545	0.2411	0.2122	0.2652	0.2334
0.260	0.1374	0.2903	0.2555	0.2420	0.2130	0.2662	0.2342
0.261	0.1380	0.2914	0.2565	0.2429	0.2138	0.2672	0.2351
0.262	0.1385	0.2924	0.2574	0.2438	0.2146	0.2681	0.2359
0.263	0.1390	0.2935	0.2584	0.2447	0.2154	0.2691	0.2368
0.264	0.1395	0.2946	0.2593	0.2456	0.2162	0.2701	0.2377
0.265	0.1400	0.2957	0.2603	0.2465	0.2170	0.2711	0.2385
0.266	0.1405	0.2968	0.2612	0.2474	0.2178	0.2721	0.2394
0.267	0.1411	0.2978	0.2622	0.2483	0.2186	0.2731	0.2403
0.268	0.1416	0.2989	0.2631	0.2492	0.2194	0.2741	0.2412
0.269	0.1421	0.3000	0.2641	0.2502	0.2202	0.2751	0.2421
0.270	0.1426	0.3011	0.2650	0.2511	0.2210	0.2761	0.2429
0.271	0.1431	0.3022	0.2660	0.2520	0.2218	0.2771	0.2438
0.272	0.1436	0.3032	0.2669	0.2529	0.2226	0.2781	0.2447
0.273	0.1442	0.3043	0.2679	0.2538	0.2234	0.2791	0.2456
0.274	0.1447	0.3054	0.2688	0.2547	0.2242	0.2801	0.2465
0.275	0.1452	0.3065	0.2698	0.2556	0.2250	0.2811	0.2473
0.276	0.1457	0.3076	0.2707	0.2565	0.2258	0.2821	0.2482
0.277	0.1462	0.3086	0.2717	0.2574	0.2266	0.2830	0.2490
0.278	0.1467	0.3097	0.2726	0.2583	0.2274	0.2840	0.2499
0.279	0.1473	0.3108	0.2736	0.2592	0.2282	0.2850	0.2508
0.280	0.1478	0.3119	0.2745	0.2602	0.2290	0.2861	0.2517
0.281	0.1483	0.3130	0.2755	0.2611	0.2298	0.2871	0.2526
0.282	0.1488	0.3140	0.2764	0.2620	0.2306	0.2880	0.2534
0.283	0.1493	0.3151	0.2774	0.2629	0.2314	0.2890	0.2543
0.284	0.1498	0.3162	0.2783	0.2638	0.2322	0.2900	0.2552
0.285	0.1504	0.3173	0.2793	0.2647	0.2330	0.2910	0.2561
0.286	0.1509	0.3184	0.2802	0.2656	0.2338	0.2920	0.2570
0.287	0.1514	0.3194	0.2812	0.2665	0.2346	0.2930	0.2578
0.288	0.1519	0.3205	0.2821	0.2674	0.2354	0.2940	0.2587
0.289	0.1524	0.3216	0.2831	0.2683	0.2362	0.2950	0.2596
0.290	0.1529	0.3227	0.2840	0.2693	0.2370	0.2960	0.2605
0.291	0.1535	0.3238	0.2850	0.2702	0.2378	0.2970	0.2614
0.292	0.1540	0.3248	0.2859	0.2711	0.2386	0.2980	0.2622
0.293	0.1545	0.3259	0.2868	0.2720	0.2394	0.2990	0.2631
0.294	0.1550	0.3270	0.2878	0.2729	0.2402	0.3000	0.2640
0.295	0.1555	0.3281	0.2887	0.2738	0.2410	0.3010	0.2649
0.296	0.1560	0.3292	0.2897	0.2747	0.2418	0.3020	0.2658
0.297	0.1566	0.3302	0.2906	0.2756	0.2426	0.3030	0.2666
0.298	0.1571	0.3313	0.2916	0.2765	0.2434	0.3040	0.2675
0.299	0.1576	0.3324	0.2925	0.2774	0.2442	0.3050	0.2684
0.300	0.1581	0.3335	0.2935	0.2784	0.2450	0.3060	0.2693

3 *Densities of solutions of cane sugar at 20°C.*

PER CENT SUGAR	TENTHS OF PER CENT					PER CENT SUGAR
	0	1	2	3	4	
0	0.998234	0.998622	0.999010	0.999398	0.999786	0
1	1,002120	1.002509	1.002897	1.003286	1.003675	1
2	1.006015	1.006405	1.006796	1.007188	1.007580	2
3	1.009934	1.010327	1.010721	1.011115	1.011510	3
4	1.013881	1.014277	1.014673	1.015070	1.015467	4
5	1.017854	1.018253	1.018652	1.019052	1.019451	5
6	1.021855	1.022257	1.022659	1.023061	1.023463	6
7	1.025885	1.026289	1.026694	1.027099	1.027504	7
8	1.029942	1.030349	1.030757	1.031165	1.031573	8
9	1.034029	1.034439	1.034850	1.035260	1.035671	9
10	1.038143	1.038556	1.038970	1.039383	1.039797	10
11	1.042288	1.042704	1.043121	1.043537	1.043954	11
12	1.046462	1.046881	1.047300	1.047720	1.048140	12
13	1.050665	1.051087	1.051510	1.051933	1.052356	13
14	1.054900	1.055325	1.055751	1.056176	1.056602	14
15	1.059165	1.059593	1.060022	1.060451	1.060880	15
16	1.063460	1.063892	1.064324	1.064756	1.065188	16
17	1.067789	1.068223	1.068658	1.069093	1.069529	17
18	1.072147	1.072585	1.073023	1.073461	1.073900	18
19	1.076537	1.076978	1.077419	1.077860	1.078302	19
20	1.080959	1.081403	1.081848	1.082292	1.082737	20
21	1.085414	1.085861	1.086309	1.086757	1.087205	21
22	1.089900	1.090351	1.090802	1.091253	1.091704	22
23	1.094420	1.094874	1.095328	1.095782	1.096236	23
24	1.098971	1.099428	1.099886	1.100344	1.100802	24
25	1.103557	1.104017	1.104478	1.104938	1.105400	25
26	1.108175	1.108639	1.109103	1.109568	1.110033	26
27	1.112828	1.113295	1.113763	1.114229	1.114697	27
28	1.117512	1.117982	1.118453	1.118923	1.119395	28
29	1.122231	1.122705	1.123179	1.123653	1.124128	29
30	1.126984	1.127461	1.127939	1.128417	1.128896	30
31	1.131773	1.132254	1.132735	1.133216	1.133698	31
32	1.136596	1.137080	1.137565	1.138049	1.138534	32
33	1.141453	1.141941	1.142429	1.142916	1.143405	33
34	1.146345	1.146836	1.147328	1.147820	1.148313	34
35	1.151275	1.151770	1.152265	1.152760	1.153256	35
36	1.156238	1.156736	1.157235	1.157733	1.158233	36
37	1.161236	1.161738	1.162240	1.162742	1.163245	37
38	1.166269	1.166775	1.167281	1.167786	1.168293	38
39	1.171340	1.171849	1.172359	1.172869	1.173379	39
40	1.176447	1.176960	1.177473	1.177987	1.178501	40
41	1.181592	1.182108	1.182625	1.183142	1.183660	41
42	1.186773	1.187293	1.187814	1.188335	1.188856	42
43	1.191993	1.192517	1.193041	1.193565	1.194090	43
44	1.197247	1.197775	1.198303	1.198832	1.199360	44
45	1,202540	1,203071	1.203603	1,204136	1,204668	45
46	1.207870	1.208405	1.208940	1.209477	1.210013	46
47	1.213238	1.213777	1.214317	1.214856	1.215395	47
48	1.218643	1.219185	1.219729	1.220272	1.220815	48
49	1.224086	1.224632	1.225180	1.225727	1.226274	49

3 *Densities of solutions of cane sugar at 20°C.*—Continued.

PER CENT SUGAR	TENTHS OF PER CENT					PER CENT SUGAR
	5	6	7	8	9	
0	1.000174	1.000563	1.000952	1.001342	1.001731	0
1	1.004064	1.004453	1.004844	1.005234	1.005624	1
2	1.007972	1.008363	1.008755	1.009148	1.009541	2
3	1.011904	1.012298	1.012694	1.013089	1.013485	3
4	1.015864	1.016261	1.016659	1.017058	1.017456	4
5	1.019851	1.020251	1.020651	1.021053	1.021454	5
6	1.023867	1.024270	1.024673	1.025077	1.025481	6
7	1.027910	1.028316	1.028722	1.029128	1.029535	7
8	1.031982	1.032391	1.032800	1.033209	1.033619	8
9	1.036082	1.036494	1.036906	1.037318	1.037730	9
10	1.040212	1.040626	1.041041	1.041456	1.041872	10
11	1.044370	1.044788	1.045206	1.045625	1.046043	11
12	1.048559	1.048980	1.049401	1.049822	1.050243	12
13	1.052778	1.053202	1.053626	1.054050	1.054475	13
14	1.057029	1.057455	1.057882	1.058310	1.058737	14
15	1.061308	1.061738	1.062168	1.062598	1.063029	15
16	1.065621	1.066054	1.066487	1.066921	1.067355	16
17	1.069964	1.070400	1.070836	1.071273	1.071710	17
18	1.074338	1.074777	1.075217	1.075657	1.076097	18
19	1.078744	1.079187	1.079629	1.080072	1.080515	19
20	1.083182	1.083628	1.084074	1.084520	1.084967	20
21	1.087652	1.088101	1.088550	1.089000	1.089450	21
22	1.092155	1.092607	1.093060	1.093513	1.093966	22
23	1.096691	1.097147	1.097603	1.098058	1.098514	23
24	1.101259	1.101718	1.102177	1.102637	1.103097	24
25	1.105862	1.106324	1.106786	1.107248	1.107711	25
26	1.110497	1.110963	1.111429	1.111895	1.112361	26
27	1.115166	1.115635	1.116104	1.116572	1.117042	27
28	1.119867	1.120339	1.120812	1.121284	1.121757	28
29	1.124603	1.125079	1,125555	1.126030	1.126507	29
30	1.129374	1.129853	1.130332	1.130812	1.131292	30
31	1.134180	1.134663	1.135146	1.135628	1.136112	31
32	1.139020	1.139506	1.139993	1.140479	1.140966	32
33	1.143894	1.144384	1.144874	1.145363	1.145854	33
34	1.148805	1.149298	1.149792	1.150286	1.150780	34
35	1.153752	1.154249	1.154746	1.155242	1.155740	35
36	1.158733	1.159233	1.159733	1.160233	1.160734	36
37	1.163748	1.164252	1.164756	1.165259	1.165764	37
38	1.168800	1.169307	1.169815	1.170322	1.170831	38
39	1.173889	1.174400	1.174911	1.175423	1.175935	39
40	1.179014	1.179527	1.180044	1.180560	1.181076	40
41	1.184178	1.184696	1.185215	1.185734	1.186253	41
42	1.189379	1.189901	1.190423	1.190946	1.191469	42
43	1.194616	1.195141	1.195667	1.196193	1.196720	43
44	1.199890	1.200420	1.200950	1.201480	1.202010	44
45	1,205200	1.205733	1.206266	1,206801	1,207335	45
46	1.210549	1.211086	1.211623	1.212162	1.212700	46
47	1.215936	1.216476	1.217017	1.217559	1,218101	47
48	1.221360	1.221904	1.222449	1.222995	1.223540	48
49	1.226823	1.227371	1.227919	1.228469	1.229018	49

3 *Densities of solutions of cane sugar at 20°C.*—Continued.

PER CENT SUGAR	TENTHS OF PER CENT					PER CENT U.
	0	1	2	3	4	
50	1.229567	1.230117	1.230668	1.231219	1.231770	50
51	1.235085	1.235639	1.236194	1.236748	1.237303	51
52	1.240641	1.241198	1.241757	1.242315	1.242873	52
53	1.246234	1.246795	1.247358	1.247920	1.248482	53
54	1.251866	1.252431	1.252997	1.253563	1.254129	54
55	1.257535	1.258104	1.258674	1.259244	1.259815	55
56	1.263243	1.263816	1.264390	1.264963	1.265537	56
57	1.268989	1.269565	1.270143	1.270720	1.271299	57
58	1.274774	1.275354	1.275936	1.276517	1.277098	58
59	1.280595	1.281179	1.281764	1.282349	1.282935	59
60	1.286456	1.287044	1.287633	1.288222	1.288811	60
61	1.292354	1.292946	1.293539	1.294131	1.294725	61
62	1.298291	1.298886	1.299483	1.300079	1.300677	62
63	1.304267	1.304867	1.305467	1.306068	1.306669	63
64	1.310282	1.310885	1.311489	1,312093	1.312699	64
65	1.316334	1.316941	1.317549	1.318157	1.318766	65
66	1.322425	1.323036	1.323648	1.324259	1.324872	66
67	1.328554	1.329170	1.329785	1.330401	1.331017	67
68	1.334722	1.335342	1.335961	1.336581	1.337200	68
69	1.340928	1.341551	1.342174	1.342798	1.343421	69
70	1.347174	1.347801	1.348427	1.349055	1.349682	70
71	1.353456	1.354087	1.354717	1.355349	1.355980	71
72	1.359778	1.360413	1.361047	1.361682	1.362317	72
73	1.366139	1.366777	1.367415	1.368054	1.368693	73
74	1.372536	1.373178	1.373820	1.374463	1.375105	74
75	1.378971	1.379617	1.380262	1.380909	1.381555	75
76	1.385446	1.386096	1.386745	1.387396	1.388045	76
77	1.391956	1.392610	1.393263	1.393917	1.394571	77
78	1.398505	1.399162	1.399819	1.400477	1.401134	78
79	1.405091	1.405752	1.406412	1.407074	1.407735	79
80	1.411715	1.412380	1.413044	1.413709	1.414374	80
81	1.418374	1.419043	1.419711	1.420380	1.421049	81
82	1.425072	1.425744	1.426416	1.427089	1.427761	82
83	1.431807	1.432483	1.433158	1.433835	1.434511	83
84	1.438579	1.439259	1.439938	1.440619	1.441299	84
85	1.445388	1.446071	1.446754	1.447438	1.448121	85
86	1.452232	1.452919	1.453605	1.454292	1.454980	86
87	1.459114	1.459805	1.460495	1.461186	1,461877	87
88	1.466032	1.466726	1.467420	1.468115	1.468810	88
89	1.472986	1.473684	1.474381	1.475080	1.475779	89
90	1.479976	1.480677	1.481378	1.482080	1.482782	90
91	1.487002	1.487707	1.488411	1.489117	1.489823	91
92	1.494063	1.494771	1.495479	1.496188	1.496897	92
93	1.501158	1.501870	1.502582	1.503293	1.504006	93
94	1.508289	1.509004	1.509720	1.510435	1.511151	94
95	1.515455	1.516174	1.516893	1.517612	1.518332	95
96	1.522656	1.523378	1.524100	1.524823	1.525546	96
97	1.529891	1.530616	1.531342	1.532068	1.532794	97
98	1.537161	1.537889	1.538618	1.539347	1.540076	98
99	1.544462	1.545194	1.545926	1.546659	1.547392	99
100	1.551800					100

3 *Densities of solutions of cane sugar at 20°C.*—Concluded.

PER CENT SUGAR	TENTHS OF PER CENT					PER CENT SUGAR
	5	6	7	8	9	
50	1.232322	1.232874	1.233426	1.233979	1.234532	50
51	1.237859	1.238414	1.238970	1.239527	1.240084	51
52	1.243433	1.243992	1.244552	1.245113	1.245673	52
53	1.249046	1.249609	1.250172	1.250737	1.251301	53
54	1.254697	1.255264	1.255831	1.256400	1.256967	54
55	1.260385	1.260955	1.261527	1.262099	1.262671	55
56	1.266112	1.266686	1.267261	1.267837	1.268413	56
57	1.271877	1.272455	1.273035	1.273614	1.274194	57
58	1.277680	1.278262	1.278844	1.279428	1.280011	58
59	1.283521	1.284107	1.284694	1.285281	1.285869	59
60	1.289401	1.289991	1.290581	1.291172	1.291763	60
61	1.295318	1.295911	1.296506	1.297100	1.297696	61
62	1.301274	1.301871	1.302470	1.303068	1.303668	62
63	1.307271	1.307872	1.308475	1.309077	1.309680	63
64	1.313304	1.313909	1,314515	1.315121	1.315728	64
65	1.319374	1.319983	1.320593	1.321203	1.321814	65
66	1.325484	1.326097	1.326711	1.327325	1.327940	66
67	1.331633	1.332250	1.332868	1.333485	1.334103	67
68	1.337821	1.338441	1.339063	1.339684	1.340306	68
69	1.344046	1.344671	1.345296	1.345922	1.346547	69
70	1.350311	1.350939	1.351568	1.352197	1.352827	70
71	1.356612	1.357245	1.357877	1.358511	1.359144	71
72	1.362953	1.363590	1.364226	1.364864	1.365501	72
73	1.369333	1.369973	1.370613	1.371254	1.371894	73
74	1.375749	1.376392	1.377036	1.377680	1.378326	74
75	1.382203	1.382851	1.383499	1.384148	1.384796	75
76	1.388696	1.389347	1.389999	1.390651	1.391303	76
77	1.395226	1.395881	1.396536	1.397192	1.397848	77
78	1.401793	1.402452	1.403111	1.403771	1.404430	78
79	1.408398	1.409061	1.409723	1.410387	1.411051	79
80	1.415040	1.415706	1.416373	1.417039	1.417707	80
81	1.421719	1.422390	1.423059	1.423730	1.424400	81
82	1.428435	1.429109	1.429782	1.430457	1.431131	82
83	1.435188	1.435866	1.436543	1.437222	1.437900	83
84	1.441980	1.442661	1.443342	1.444024	1.444705	84
85	1.448806	1.449491	1.450175	1.450860	1.451545	85
86	1.455668	1.456357	1.457045	1.457735	1.458424	86
87	1.462568	1.463260	1.463953	1.464645	1.465338	87
88	1.469504	1.470200	1.470896	1.471592	1.472289	88
89	1.476477	1.477176	1.477876	1.478575	1.479275	89
90	1.483484	1.484187	1.484890	1.485593	1.486297	90
91	1.490528	1.491234	1.491941	1.492647	1.493355	91
92	1.497606	1.498316	1.499026	1.499736	1.500447	92
93	1.504719	1.505432	1.506146	1.506859	1.507574	93
94	1.511868	1.512585	1.513302	1.514019	1.514737	94
95	1.519051	1.519771	1.520492	1.521212	1.521934	95
96	1.526269	1.526993	1.527717	1.528441	1.529166	96
97	1.533521	1.534248	1.534976	1.535791	1.536432	97
98	1.540806	1.541536	1.542267	1.542998	1.543730	98
99	1.548127	1.548861	1.549595	1.550329	1.551064	99
100						100

4 *Corrections to be applied to results obtained by 3 when the specific gravity is obtained at temperatures other than 20°C.*

(This table is calculated using the data on thermal expansion of sugar solutions by Plato, assuming the instrument to be of Jena 16^{III} glass. The table should be used with caution and only for approximate results when the temperature differs much from the standard temperature or from the temperature of the surrounding air.)

TEMPERATURE IN DEGREES CENTIGRADE	OBSERVED PER CENT OF SUGAR													
	0	5	10	15	20	25	30	35	40	45	50	55	60	70
	Subtract—													
0	0.30	0.49	0.65	0.77	0.89	0.99	1.08	1.16	1.24	1.31	1.37	1.41	1.44	1.49
5	0.36	0.47	0.56	0.65	0.73	0.80	0.86	0.91	0.97	1.01	1.05	1.08	1.10	1.14
10	0.32	0.38	0.43	0.48	0.52	0.57	0.60	0.64	0.67	0.70	0.72	0.74	0.75	0.77
11	0.31	0.35	0.40	0.44	0.48	0.51	0.55	0.58	0.60	0.63	0.65	0.66	0.68	0.70
12	0.29	0.32	0.36	0.40	0.43	0.46	0.50	0.52	0.54	0.56	0.58	0.59	0.60	0.62
13	0.26	0.29	0.32	0.35	0.38	0.41	0.44	0.46	0.48	0.49	0.51	0.52	0.53	0.55
14	0.24	0.26	0.29	0.31	0.34	0.36	0.38	0.40	0.41	0.42	0.44	0.45	0.46	0.47
15	0.20	0.22	0.24	0.26	0.28	0.30	0.32	0.33	0.34	0.36	0.36	0.37	0.38	0.39
16	0.17	0.18	0.20	0.22	0.23	0.25	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32
17	0.13	0.14	0.15	0.16	0.18	0.19	0.20	0.20	0.21	0.21	0.22	0.23	0.23	0.24
18	0.09	0.10	0.10	0.11	0.12	0.13	0.13	0.14	0.14	0.14	0.15	0.15	0.15	0.16
19	0.05	0.05	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08
17.5	0.11	0.12	0.12	0.14	0.15	0.16	0.16	0.17	0.17	0.18	0.18	0.19	0.19	0.20
15.56 (60°F.)	0.18	0.20	0.22	0.24	0.26	0.28	0.29	0.30	0.30	0.32	0.33	0.33	0.34	0.34
	Add—													
21	0.04	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.09
22	0.10	0.10	0.11	0.12	0.12	0.13	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.16
23	0.16	0.16	0.17	0.17	0.19	0.20	0.21	0.21	0.22	0.23	0.24	0.24	0.24	0.24
24	0.21	0.22	0.23	0.24	0.26	0.27	0.28	0.29	0.30	0.31	0.32	0.32	0.32	0.32
25	0.27	0.28	0.30	0.31	0.32	0.34	0.35	0.36	0.38	0.38	0.39	0.39	0.40	0.39
26	0.33	0.34	0.36	0.37	0.40	0.40	0.42	0.44	0.46	0.47	0.47	0.48	0.48	0.48
27	0.40	0.41	0.42	0.44	0.46	0.48	0.50	0.52	0.54	0.54	0.55	0.56	0.56	0.56
28	0.46	0.47	0.49	0.51	0.54	0.56	0.58	0.60	0.61	0.62	0.63	0.64	0.64	0.64
29	0.54	0.55	0.56	0.59	0.61	0.63	0.66	0.68	0.70	0.70	0.71	0.72	0.72	0.72
30	0.61	0.62	0.63	0.66	0.68	0.71	0.73	0.76	0.78	0.78	0.79	0.80	0.80	0.81
35	0.99	1.01	1.02	1.06	1.10	1.13	1.16	1.18	1.20	1.21	1.22	1.22	1.23	1.22
40	1.42	1.45	1.47	1.51	1.54	1.57	1.60	1.62	1.64	1.65	1.65	1.65	1.66	1.65
45	1.91	1.94	1.96	2.00	2.03	2.05	2.07	2.09	2.10	2.10	2.10	2.10	2.10	2.08
50	2.46	2.48	2.50	2.53	2.56	2.57	2.58	2.59	2.59	2.58	2.58	2.57	2.56	2.52
55	3.05	3.07	3.09	3.12	3.12	3.12	3.12	3.11	3.10	3.08	3.07	3.05	3.03	2.97
60	3.69	3.72	3.73	3.73	3.72	3.70	3.67	3.65	3.62	3.60	3.57	3.54	3.50	3.43
27.5	0.43	0.44	0.46	0.48	0.50	0.52	0.54	0.56	0.58	0.58	0.59	0.60	0.60	0.60

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1. Wiss. Abh. der Kaiserlichen Normal-Eichungs-Kommission, 2, p. 140; 1900.

5

Geerligs' Table.

For dry substance in sugar-house products by the Abbe refractometer, at 28°C.

INDEX	PER CENT DRY SUBSTANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS[a]	INDEX	PER CENT DRY SUBSTANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS[a]	INDEX	PER CENT DRY SUBSTANCE	DECIMALS TO BE ADDED FOR FRACTIONAL READINGS[a]
1.3335	1	0.0001=0.05	1.3484	11	0.0001=0.05	1.3746	27	0.0001=0.05
1.3349	2	0.0002=0.1	1.3500	12	0.0002=0.1	1.3764	28	0.0002=0.1
1.3364	3	0.0003=0.2	1.3516	13	0.0003=0.2	1.3782	29	0.0003=0.15
1.3379	4	0.0004=0.25	1.3530	14	0.0004=0.25	1,3800	30	0.0004=0.2
1.3394	5	0.0005=0.3	1.3546	15	0.0005=0.3	1,3818	31	0.0005=0.25
1.3409	6	0.0006=0.4	1.3562	16	0.0006=0.4	1,3836	32	0.0006=0.3
1.3424	7	0.0007=0.5	1.3578	17	0.0007=0.45	1.3854	33	0.0007=0.35
1.3439	8	0.0008=0.6	1.3594	18	0.0008=0.5	1.3872	34	0.0008=0.4
1.3454	9	0.0009=0.7	1.3611	19	0.0009=0.6	1.3890	35	0.0009=0.45
1.3469	10	0.0010=0.75	1.3627	20	0.0010=0.65	1.3909	36	0.0010=0.5
		0.0011=0.8	1.3644	21	0.0011=0.7	1.3928	37	0.0011=0.55
		0.0012=0.8	1,3661	22	0.0012=0.75	1.3947	38	0.0012=0.6
		0.0013=0.85	1.3678	23	0.0013=0.8	1.3966	39	0.0013=0.65
		0.0014=0.9	1.3695	24	0.0014=0.85	1,3984	40	0.0014=0.7
		0.0015=1.0	1.3712	25	0.0015=0.9	1.4003	41	0.0015=0.75
			1.3729	26	16=0.95			0.0016=0.8
								0.0017=0.85
								0.0018=0.9
								0.0019=0.95
								0.0020=1.0
								0.0021=1.0
1.4023	42	0.0001=0.05	1.4292	55	0.0001=0.05	1.4711	73	0.0001=0.0
1.4043	43	0.0002=0.1	1.4314	56	0.0002=0.1	1.4736	74	0.0002=0.05
1.4063	44	0.0003=0.15	1.4337	57	0.0003=0.1	1.4761	75	0.0003=0.1
1.4083	45	0.0004=0.2	1.4359	58	0.0004=0.15	1.4786	76	0.0004=0.15
1.4104	46	0.0005=0.25	1.4382	59	0.0005=0.2	1.4811	77	0.0005=0.2
1.4124	47	0.0006=0.3	1.4405	60	0.0006=0.25	1.4836	78	0.0006=0.2
1.4145	48	0.0007=0.35	1.4428	61	0.0007=0.3	1.4862	79	0.0007=0.25
1.4166	49	0.0008=0.4	1.4451	62	0.0008=0.35	1.4888	80	0.0008=0.3
1.4186	50	0.0009=0.45	1.4474	63	0.0009=0.4	1.4914	81	0.0009=0.35
1.4207	51	0.0010=0.5	1.4497	64	0.0010=0.45	1.4940	82	0.0010=0.35
1.4228	52	0.0011=0.55	1.4520	65	0.0011=0.5	1.4966	83	0.0011=0.4
1.4249	53	0.0012=0.6	1.4543	66	0.0012=0.5	1.4992	84	0.0012=0.45
1.4270	54	0.0013=0.65	1.4567	67	0.0013=0.55	1.5019	85	0.0013=0.5
		0.0014=0.7	1.4591	68	0.0014=0.6	1.5046	86	0.0014=0.5
		0.0015=0.75	1,4615	69	0.0015=0.65	1.5073	87	0.0015=0.55
		0.0016=0.8	1.4639	70	0.0016=0.7	1.5100	88	0.0016=0.6
		0.0017=0.85	1.4663	71	0.0017=0.75	1.5127	89	0.0017=0.65
		0.0018=0.9	1.4687	72	0.0018=0.8	1.5155	90	0.0018=0.65
		0.0019=0.95			0.0019=0.85			0.0019=1.7
		0.0020=1.0			0.0020=0.9			0.0020=0.75
		0.0021=1.0			0.0021=0.9			0.0021=0.8
					0.0022=0.95			0.0022=0.8
					0.0023=1.0			0.0023=0.85
					0.0024=1.0			0.0024=0.9
								0.0025=0.9
								0.0026=0.95
								0.0027=1.0
								0.0028=1.0

[a] Find in the table the refractive index which is next lower than the reading actually made and note the corresponding whole number for the per cent of dry substance. Subtract the refractive index obtained from the table from the observed reading; the decimal corresponding to this difference, as given in the column so marked, is added to the whole per cent of dry substance as first obtained.

6 *Corrections for temperature to be used in conjunction with* **5**.

TEMPERATURE OF THE PRISMS IN °C.	DRY SUBSTANCE												
	0	5	10	15	20	25	30	40	50	60	70	80	90
	Subtract—												
20	0.53	0.54	0.55	0.56	0.57	0.58	0.60	0.62	0.64	0.62	0.61	0.60	0.58
21	0.46	0.47	0.48	0.49	0.50	0.51	0.52	0.54	0.56	0.54	0.53	0.52	0.50
22	0.40	0.41	0.42	0.42	0.43	0.44	0.45	0.47	0.48	0.47	0.46	0.45	0.44
23	0.33	0.33	0.34	0.35	0.36	0.37	0.38	0.39	0.40	0.39	0.38	0.38	0.38
24	0.26	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32	0.31	0.31	0.30	0.30
25	0.20	0.20	0.21	0.21	0.22	0.22	0.23	0.23	0.24	0.23	0.23	0.23	0.22
26	0.12	0.12	0.13	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.15	0.15	0.14
27	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
	Add—												
29	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
30	0.12	0.12	0.13	0.14	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.15	0.14
31	0.20	0.20	0.21	0.21	0.22	0.22	0.23	0.23	0.24	0.23	0.23	0.23	0.22
32	0.26	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32	0.31	0.31	0.30	0.30
33	0.33	0.33	0.34	0.35	0.36	0.37	0.38	0.39	0.40	0.39	0.38	0.38	0.38
34	0.40	0.41	0.42	0.42	0.43	0.44	0.45	0.47	0.48	0.47	0.46	0.45	0.44
35	0.46	0.47	0.48	0.49	0.50	0.51	0.52	0.54	0.56	0.54	0.53	0.52	0.50

7

Alcohol Table.

(Calculated by U. S. Bureau of Standards from its experimental results.)

For calculating the percentages of alcohol in mixtures of ethyl alcohol and water from their specific gravities.

SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL			SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL			SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.99823	**0.00**	*0.00*	0.00	0.99492	**2.25**	*1.79*	1.78	0.99174	**4.50**	*3.58*	3.55
0.99815	**0.05**	*0.04*	0.04	0.99485	**2.30**	*1.82*	1.81	0.99168	**4.55**	*3.62*	3.59
0.99808	**0.10**	*0.08*	0.08	0.99477	**2.35**	*1.86*	1.85	0.99161	**4.60**	*3.66*	3.63
0.99800	**0.15**	*0.12*	0.12	0.99470	**2.40**	*1.90*	1.89	0.99154	**4.65**	*3.70*	3.67
0.99793	**0.20**	*0.16*	0.16	0.99463	**2.45**	*1.94*	1.93	0.99147	**4.70**	*3.74*	3.71
0.99785	**0.25**	*0.20*	0.20	0.99456	**2.50**	*1.98*	1.97	0.99140	**4.75**	*3.78*	3.75
0.99778	**0.30**	*0.24*	0.24	0.99449	**2.55**	*2.02*	2.01	0.99133	**4.80**	*3.82*	3.79
0.99770	**0.35**	*0.28*	0.28	0.99442	**2.60**	*2.06*	2.05	0.99127	**4.85**	*3.86*	3.83
0.99763	**0.40**	*0.32*	0.32	0.99434	**2.65**	*2.10*	2.09	0.99120	**4.90**	*3.90*	3.87
0.99755	**0.45**	*0.36*	0.36	0.99427	**2.70**	*2.14*	2.13	0.99113	**4.95**	*3.94*	3.91
0.99748	**0.50**	*0.40*	0.40	0.99420	**2.75**	*2.18*	2.17	0.99106	**5.00**	*3.98*	3.95
0.99741	**0.55**	*0.44*	0.44	0.99413	**2.80**	*2.22*	2.21	0.99100	**5.05**	*4.02*	3.99
0.99734	**0.60**	*0.47*	0.47	0.99405	**2.85**	*2.26*	2.25	0.99093	**5.10**	*4.06*	4.03
0.99726	**0.65**	*0.51*	0.51	0.99398	**2.90**	*2.30*	2.29	0.99087	**5.15**	*4.10*	4.07
0.99719	**0.70**	*0.55*	0.55	0.99391	**2.95**	*2.34*	2.33	0.99080	**5.20**	*4.14*	4.10
0.99711	**0.75**	*0.59*	0.59	0.99384	**3.00**	*2.38*	2.37	0.99073	**5.25**	*4.18*	4.14
0.99704	**0.80**	*0.63*	0.63	0.99377	**3.05**	*2.42*	2.41	0.99066	**5.30**	*4.22*	4.18
0.99697	**0.85**	*0.67*	0.67	0.99370	**3.10**	*2.46*	2.45	0.99060	**5.35**	*4.26*	4.22
0.99690	**0.90**	*0.71*	0.71	0.99362	**3.15**	*2.50*	2.49	0.99053	**5.40**	*4.30*	4.26
0.99682	**0.95**	*0.75*	0.75	0.99355	**3.20**	*2.54*	2.53	0.99047	**5.45**	*4.34*	4.30
0.99675	**1.00**	*0.79*	0.79	0.99348	**3.25**	*2.58*	2.57	0.99040	**5.50**	*4.38*	4.34
0.99667	**1.05**	*0.83*	0.83	0.99341	**3.30**	*2.62*	2.60	0.99033	**5.55**	*4.42*	4.38
0.99660	**1.10**	*0.87*	0.87	0.99334	**3.35**	*2.66*	2.64	0.99026	**5.60**	*4.46*	4.42
0.99652	**1.15**	*0.91*	0.91	0.99327	**3.40**	*2.70*	2.68	0.99020	**5.65**	*4.50*	4.46
0.99645	**1.20**	*0.95*	0.95	0.99320	**3.45**	*2.74*	2.72	0.99013	**5.70**	*4.54*	4.50
0.99638	**1.25**	*0.99*	0.99	0.99313	**3.50**	*2.78*	2.76	0.99006	**5.75**	*4.58*	4.54
0.99631	**1.30**	*1.03*	1.03	0.99306	**3.55**	*2.82*	2.80	0.98999	**5.80**	*4.62*	4.58
0.99623	**1.35**	*1.07*	1.07	0.99299	**3.60**	*2.86*	2.84	0.98993	**5.85**	*4.66*	4.62
0.99616	**1.40**	*1.11*	1.11	0.99292	**3.65**	*2.90*	2.88	0.98986	**5.90**	*4.70*	4.66
0.99608	**1.45**	*1.15*	1.15	0.99285	**3.70**	*2.94*	2.92	0.98980	**5.95**	*4.74*	4.70
0.99601	**1.50**	*1.19*	1.19	0.99278	**3.75**	*2.98*	2.96	0.98973	**6.00**	*4.78*	4.74
0.99594	**1.55**	*1.23*	1.23	0.99271	**3.80**	*3.02*	3.00	0.98967	**6.05**	*4.82*	4.78
0.99587	**1.60**	*1.27*	1.26	0.99264	**3.85**	*3.06*	3.04	0.98960	**6.10**	*4.87*	4.82
0.99579	**1.65**	*1.31*	1.30	0.99257	**3.90**	*3.10*	3.08	0.98954	**6.15**	*4.91*	4.86
0.99572	**1.70**	*1.35*	1.34	0.99250	**3.95**	*3.14*	3.12	0.98947	**6.20**	*4.95*	4.89
0.99564	**1.75**	*1.39*	1.38	0.99243	**4.00**	*3.18*	3.16	0.98941	**6.25**	*4.99*	4.93
0.99557	**1.80**	*1.43*	1.42	0.99236	**4.05**	*3.22*	3.20	0.98934	**6.30**	*5.03*	4.97
0.99550	**1.85**	*1.47*	1.46	0.99229	**4.10**	*3.26*	3.24	0.98928	**6.35**	*5.07*	5.01
0.99543	**1.90**	*1.51*	1.50	0.99222	**4.15**	*3.30*	3.28	0.98921	**6.40**	*5.11*	5.05
0.99535	**1.95**	*1.55*	1.54	0.99215	**4.20**	*3.34*	3.32	0.98915	**6.45**	*5.15*	5.09
0.99528	**2.00**	*1.59*	1.58	0.99208	**4.25**	*3.38*	3.36	0.98908	**6.50**	*5.19*	5.13
0.99520	**2.05**	*1.63*	1.62	0.99201	**4.30**	*3.42*	3.39	0.98902	**6.55**	*5.23*	5.17
0.99513	**2.10**	*1.67*	1.66	0.99195	**4.35**	*3.46*	3.43	0.98895	**6.60**	*5.27*	5.21
0.99506	**2.15**	*1.71*	1.70	0.99188	**4.40**	*3.50*	3.47	0.98889	**6.65**	*5.31*	5.25
0.99499	**2.20**	*1.75*	1.74	0.99181	**4.45**	*3.54*	3.51	0.98882	**6.70**	*5.35*	5.29

7

Alcohol Table.—Continued

Specific gravity 20° C./4°	Alcohol			Specific gravity 20° C./4°	Alcohol			Specific gravity 20° C./4°	Alcohol		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.98876	6.75	5.39	5.33	0.98566	9.25	7.41	7.30	0.98267	11.75	9.44	9.28
0.98870	6.80	5.43	5.37	0.98560	9.30	7.45	7.34	0.98261	11.80	9.48	9.31
0.98864	6.85	5.47	5.41	0.98554	9.35	7.49	7.38	0.98255	11.85	9.52	9.35
0.98857	6.90	5.51	5.45	0.98549	9.40	7.53	7.42	0.98250	11.90	9.56	9.39
0.98851	6.95	5.55	5.49	0.98543	9.45	7.57	7.46	0.98244	11.95	9.60	9.43
0.98845	7.00	5.59	5.53	0.98537	9.50	7.61	7.50	0.98238	12.00	9.64	9.47
0.98839	7.05	5.63	5.57	0.98531	9.55	7.65	7.54	0.98232	12.05	9.68	9.51
0.98832	7.10	5.67	5.60	0.98524	9.60	7.69	7.58	0.98226	12.10	9.72	9.55
0.98826	7.15	5.71	5.64	0.98518	9.65	7.73	7.62	0.98220	12.15	9.76	9.59
0.98820	7.20	5.75	5.68	0.98512	9.70	7.77	7.66	0.98214	12.20	9.80	9.63
0.98813	7.25	5.79	5.72	0.98506	9.75	7.81	7.70	0.98208	12.25	9.84	9.67
0.98806	7.30	5.83	5.76	0.98501	9.80	7.85	7.73	0.98203	12.30	9.89	9.71
0.98800	7.35	5.87	5.80	0.98495	9.85	7.89	7.77	0.98197	12.35	9.93	9.75
0.98794	7.40	5.91	5.84	0.98488	9.90	7.93	7.81	0.98191	12.40	9.97	9.79
0.98788	7.45	5.95	5.88	0.98482	9.95	7.97	7.85	0.98185	12.45	10.01	9.83
0.98781	7.50	5.99	5.92	0.98476	10.00	8.02	7.89	0.98180	12.50	10.05	9.87
0.98775	7.55	6.03	5.96	0.98470	10.05	8.06	7.93	0.98174	12.55	10.09	9.91
0.98769	7.60	6.07	6.00	0.98463	10.10	8.10	7.97	0.98168	12.60	10.13	9.95
0.98763	7.65	6.11	6.04	0.98457	10.15	8.14	8.01	0.98162	12.65	10.17	9.99
0.98756	7.70	6.15	6.08	0.98452	10.20	8.18	8.05	0.98156	12.70	10.21	10.03
0.98750	7.75	6.19	6.12	0.98446	10.25	8.22	8.09	0.98150	12.75	10.25	10.07
0.98744	7.80	6.24	6.16	0.98441	10.30	8.26	8.13	0.98145	12.80	10.29	10.10
0.98738	7.85	6.28	6.20	0.98435	10.35	8.30	8.17	0.98139	12.85	10.33	10.14
0.98731	7.90	6.32	6.24	0.98428	10.40	8.34	8.21	0.98132	12.90	10.38	10.18
0.98725	7.95	6.36	6.28	0.98422	10.45	8.38	8.25	0.98127	12.95	10.42	10.22
0.98718	8.00	6.40	6.32	0.98416	10.50	8.42	8.29	0.98122	13.00	10.46	10.26
0.98712	8.05	6.44	6.36	0.98410	10.55	8.46	8.33	0.98116	13.05	10.50	10.30
0.98706	8.10	6.48	6.39	0.98404	10.60	8.50	8.37	0.98111	13.10	10.54	10.34
0.98700	8.15	6.52	6.43	0.98398	10.65	8.54	8.41	0.98105	13.15	10.58	10.38
0.98694	8.20	6.56	6.47	0.98391	10.70	8.58	8.45	0.98100	13.20	10.62	10.42
0.98688	8.25	6.60	6.51	0.98385	10.75	8.62	8.49	0.98094	13.25	10.66	10.46
0.98682	8.30	6.64	6.55	0.98379	10.80	8.66	8.52	0.98089	13.30	10.70	10.50
0.98676	8.35	6.68	6.59	0.98373	10.85	8.70	8.56	0.98083	13.35	10.74	10.54
0.98670	8.40	6.72	6.63	0.98368	10.90	8.75	8.60	0.98077	13.40	10.78	10.58
0.98664	8.45	6.76	6.67	0.98362	10.95	8.79	8.64	0.98071	13.45	10.82	10.62
0.98658	8.50	6.80	6.71	0.98356	11.00	8.83	8.68	0.98066	13.50	10.86	10.66
0.98652	8.55	6.84	6.75	0.98350	11.05	8.87	8.72	0.98060	13.55	10.90	10.70
0.98646	8.60	6.88	6.79	0.98344	11.10	8.91	8.76	0.98054	13.60	10.95	10.74
0.98640	8.65	6.92	6.83	0.98338	11.15	8.95	8.80	0.98048	13.65	10.99	10.78
0.98633	8.70	6.96	6.87	0.98332	11.20	8.99	8.84	0.98043	13.70	11.03	10.81
0.98627	8.75	7.00	6.91	0.98326	11.25	9.03	8.88	0.98037	13.75	11.07	10.85
0.98620	8.80	7.04	6.95	0.98320	11.30	9.07	8.92	0.98031	13.80	11.11	10.89
0.98614	8.85	7.08	6.99	0.98314	11.35	9.11	8.96	0.98025	13.85	11.15	10.93
0.98608	8.90	7.12	7.03	0.98308	11.40	9.15	9.00	0.98020	13.90	11.19	10.97
0.98602	8.95	7.16	7.07	0.98302	11.45	9.19	9.04	0.98014	13.95	11.23	11.01
0.98596	9.00	7.20	7.10	0.98296	11.50	9.23	9.08	0.98009	14.00	11.28	11.05
0.98590	9.05	7.24	7.14	0.98290	11.55	9.27	9.12	0.98003	14.05	11.32	11.09
0.98584	9.10	7.29	7.18	0.98285	11.60	9.32	9.16	0.97998	14.10	11.36	11.13
0.98578	9.15	7.33	7.22	0.98279	11.65	9.36	9.20	0.97992	14.15	11.40	11.17
0.98572	9.20	7.37	7.26	0.98273	11.70	9.40	9.24	0.97986	14.20	11.44	11.21

7

Alcohol Table.—Continued.

Specific gravity 20° C./4°	Alcohol			Specific gravity 20° C./4°	Alcohol			Specific gravity 20° C./4°	Alcohol		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.97980	**14.25**	*11.48*	11.25	0.97704	**16.75**	*13.53*	13.22	0.97438	**19.25**	*15.59*	15.20
0.97975	**14.30**	*11.52*	11.29	0.97699	**16.80**	*13.57*	13.26	0.97433	**19.30**	*15.64*	15.23
0.97969	**14.35**	*11.56*	11.33	0.97694	**16.85**	*13.61*	13.30	0.97428	**19.35**	*15.68*	15.27
0.97964	**14.40**	*11.60*	11.37	0.97689	**16.90**	*13.66*	13.34	0.97423	**19.40**	*15.72*	15.31
0.97958	**14.45**	*11.64*	11.41	0.97683	**16.95**	*13.70*	13.38	0.97417	**19.45**	*15.76*	15.35
0.97953	**14.50**	*11.68*	11.44	0.97678	**17.00**	*13.74*	13.42	0.97412	**19.50**	*15.80*	15.39
0.97947	**14.55**	*11.72*	11.48	0.97672	**17.05**	*13.78*	13.46	0.97407	**19.55**	*15.84*	15.43
0.97942	**14.60**	*11.77*	11.52	0.97667	**17.10**	*13.82*	13.50	0.97402	**19.60**	*15.88*	15.47
0.97936	**14.65**	*11.81*	11.56	0.97661	**17.15**	*13.86*	13.54	0.97396	**19.65**	*15.92*	15.51
0.97930	**14.70**	*11.85*	11.60	0.97656	**17.20**	*13.90*	13.58	0.97391	**19.70**	*15.97*	15.55
0.97924	**14.75**	*11.89*	11.64	0.97650	**17.25**	*13.94*	13.62	0.97386	**19.75**	*16.01*	15.59
0.97919	**14.80**	*11.93*	11.68	0.97645	**17.30**	*13.98*	13.66	0.97381	**19.80**	*16.05*	15.63
0.97913	**14.85**	*11.97*	11.72	0.97639	**17.35**	*14.02*	13.70	0.97375	**19.85**	*16.09*	15.67
0.97908	**14.90**	*12.01*	11.76	0.97634	**17.40**	*14.07*	13.74	0.97370	**19.90**	*16.13*	15.71
0.97902	**14.95**	*12.05*	11.80	0.97629	**17.45**	*14.11*	13.78	0.97364	**19.95**	*16.17*	15.75
0.97897	**15.00**	*12.09*	11.84	0.97624	**17.50**	*14.15*	13.81	0.97359	**20.00**	*16.21*	15.79
0.97891	**15.05**	*12.13*	11.88	0.97618	**17.55**	*14.19*	13.85	0.97354	**20.05**	*16.25*	15.83
0.97885	**15.10**	*12.18*	11.92	0.97613	**17.60**	*14.23*	13.89	0.97349	**20.10**	*16.30*	15.87
0.97879	**15.15**	*12.22*	11.96	0.97607	**17.65**	*14.27*	13.93	0.97344	**20.15**	*16.34*	15.91
0.97874	**15.20**	*12.26*	12.00	0.97602	**17.70**	*14.31*	13.97	0.97339	**20.20**	*16.38*	15.95
0.97868	**15.25**	*12.30*	12.04	0.97596	**17.75**	*14.35*	14.01	0.97333	**20.25**	*16.42*	15.99
0.97863	**15.30**	*12.34*	12.08	0.97591	**17.80**	*14.40*	14.05	0.97328	**20.30**	*16.46*	16.02
0.97857	**15.35**	*12.38*	12.12	0.97586	**17.85**	*14.44*	14.09	0.97322	**20.35**	*16.50*	16.06
0.97852	**15.40**	*12.42*	12.16	0.97581	**17.90**	*14.48*	14.13	0.97317	**20.40**	*16.55*	16.10
0.97846	**15.45**	*12.46*	12.20	0.97575	**17.95**	*14.52*	14.17	0.97311	**20.45**	*16.59*	16.14
0.97841	**15.50**	*12.50*	12.23	0.97570	**18.00**	*14.56*	14.21	0.97306	**20.50**	*16.63*	16.18
0.97835	**15.55**	*12.54*	12.27	0.97564	**18.05**	*14.60*	14.25	0.97300	**20.55**	*16.67*	16.22
0.97830	**15.60**	*12.59*	12.31	0.97559	**18.10**	*14.64*	14.29	0.97295	**20.60**	*16.71*	16.26
0.97824	**15.65**	*12.63*	12.35	0.97553	**18.15**	*14.68*	14.33	0.97289	**20.65**	*16.75*	16.30
0.97819	**15.70**	*12.67*	12.39	0.97548	**18.20**	*14.73*	14.37	0.97284	**20.70**	*16.80*	16.34
0.97813	**15.75**	*12.71*	12.43	0.97542	**18.25**	*14.77*	14.41	0.97278	**20.75**	*16.84*	16.38
0.97808	**15.80**	*12.75*	12.47	0.97538	**18.30**	*14.81*	14.45	0.97273	**20.80**	*16.88*	16.42
0.97802	**15.85**	*12.79*	12.51	0.97532	**18.35**	*14.85*	14.49	0.97268	**20.85**	*16.92*	16.46
0.97797	**15.90**	*12.83*	12.55	0.97527	**18.40**	*14.89*	14.52	0.97263	**20.90**	*16.96*	16.50
0.97791	**15.95**	*12.87*	12.59	0.97522	**18.45**	*14.93*	14.56	0.97257	**20.95**	*17.00*	16.54
0.97786	**16.00**	*12.92*	12.63	0.97517	**18.50**	*14.97*	14.60	0.97252	**21.00**	*17.04*	16.58
0.97780	**16.05**	*12.96*	12.67	0.97512	**18.55**	*15.01*	14.64	0.97247	**21.05**	*17.08*	16.62
0.97775	**16.10**	*13.00*	12.71	0.97507	**18.60**	*15.06*	14.68	0.97242	**21.10**	*17.13*	16.66
0.97769	**16.15**	*13.04*	12.75	0.97501	**18.65**	*15.10*	14.72	0.97237	**21.15**	*17.17*	16.70
0.97764	**16.20**	*13.08*	12.79	0.97496	**18.70**	*15.14*	14.76	0.97232	**21.20**	*17.21*	16.73
0.97758	**16.25**	*13.12*	12.83	0.97490	**18.75**	*15.18*	14.80	0.97227	**21.25**	*17.25*	16.77
0.97753	**16.30**	*13.16*	12.87	0.97485	**18.80**	*15.22*	14.84	0.97222	**21.30**	*17.29*	16.81
0.97747	**16.35**	*13.20*	12.91	0.97479	**18.85**	*15.26*	14.88	0.97216	**21.35**	*17.33*	16.85
0.97742	**16.40**	*13.24*	12.95	0.97474	**18.90**	*15.30*	14.92	0.97210	**21.40**	*17.38*	16.89
0.97737	**16.45**	*13.28*	12.99	0.97469	**18.95**	*15.34*	14.96	0.97204	**21.45**	*17.42*	16.93
0.97732	**16.50**	*13.33*	13.02	0.97464	**19.00**	*15.39*	15.00	0.97199	**21.50**	*17.46*	16.97
0.97726	**16.55**	*13.37*	13.06	0.97459	**19.05**	*15.43*	15.04	0.97193	**21.55**	*17.50*	17.01
0.97721	**16.60**	*13.41*	13.10	0.97454	**19.10**	*15.47*	15.08	0.97188	**21.60**	*17.54*	17.05
0.97715	**16.65**	*13.45*	13.14	0.97449	**19.15**	*15.51*	15.12	0.97183	**21.65**	*17.58*	17.09
0.97710	**16.70**	*13.49*	13.18	0.97444	**19.20**	*15.55*	15.16	0.97178	**21.70**	*17.63*	17.13

7

Alcohol Table.—Continued.

Specific gravity 20° C./4°	Alcohol: Per cent by volume at 20° C.	Alcohol: Per cent by weight	Alcohol: Grams per 100 cc.	Specific gravity 20° C./4°	Alcohol: Per cent by volume at 20° C.	Alcohol: Per cent by weight	Alcohol: Grams per 100 cc.	Specific gravity 20° C./4°	Alcohol: Per cent by volume at 20° C.	Alcohol: Per cent by weight	Alcohol: Grams per 100 cc.
0.97172	21.75	*17.67*	17.17	0.96896	24.25	*19.75*	19.14	0.96612	26.75	*21.85*	21.12
0.97167	21.80	*17.71*	17.21	0.96891	24.30	*19.80*	19.18	0.96606	26.80	*21.90*	21.16
0.97161	21.85	*17.75*	17.25	0.96885	24.35	*19.84*	19.22	0.96600	26.85	*21.94*	21.20
0.97156	21.90	*17.79*	17.29	0.96880	24.40	*19.88*	19.26	0.96595	26.90	*21.98*	21.23
0.97150	21.95	*17.83*	17.33	0.96874	24.45	*19.92*	19.30	0.96589	26.95	*22.02*	21.27
0.97145	22.00	*17.88*	17.37	0.96869	24.50	*19.96*	19.34	0.96583	27.00	*22.07*	21.31
0.97139	22.05	*17.92*	17.41	0.96863	24.55	*20.00*	19.38	0.96577	27.05	*22.11*	21.35
0.97134	22.10	*17.96*	17.45	0.96857	24.60	*20.05*	19.42	0.96571	27.10	*22.15*	21.39
0.97128	22.15	*18.00*	17.49	0.96851	24.65	*20.09*	19.46	0.96565	27.15	*22.19*	21.43
0.97123	22.20	*18.04*	17.52	0.96846	24.70	*20.13*	19.50	0.96559	27.20	*22.24*	21.47
0.97118	22.25	*18.08*	17.56	0.96840	24.75	*20.17*	19.54	0.96553	27.25	*22.28*	21.51
0.97113	22.30	*18.13*	17.60	0.96835	24.80	*20.22*	19.58	0.96548	27.30	*22.32*	21.55
0.97107	22.35	*18.17*	17.64	0.96829	24.85	*20.26*	19.62	0.96542	27.35	*22.36*	21.59
0.97102	22.40	*18.21*	17.68	0.96823	24.90	*20.30*	19.66	0.96536	27.40	*22.40*	21.63
0.97096	22.45	*18.25*	17.72	0.96817	24.95	*20.34*	19.70	0.96530	27.45	*22.44*	21.67
0.97091	22.50	*18.29*	17.76	0.96812	25.00	*20.38*	19.73	0.96525	27.50	*22.49*	21.71
0.97085	22.55	*18.33*	17.80	0.96806	25.05	*20.42*	19.77	0.96519	27.55	*22.53*	21.75
0.97080	22.60	*18.38*	17.84	0.96801	25.10	*20.47*	19.81	0.96513	27.60	*22.57*	21.79
0.97074	22.65	*18.42*	17.88	0.96795	25.15	*20.51*	19.85	0.96507	27.65	*22.61*	21.83
0.97069	22.70	*18.46*	17.92	0.96789	25.20	*20.55*	19.89	0.96501	27.70	*22.66*	21.87
0.97063	22.75	*18.50*	17.96	0.96783	25.25	*20.59*	19.93	0.96495	27.75	*22.70*	21.91
0.97058	22.80	*18.54*	18.00	0.96778	25.30	*20.64*	19.97	0.96489	27.80	*22.74*	21.94
0.97052	22.85	*18.58*	18.04	0.96772	25.35	*20.68*	20.01	0.96483	27.85	*22.78*	21.98
0.97047	22.90	*18.63*	18.08	0.96766	25.40	*20.72*	20.05	0.96477	27.90	*22.83*	22.02
0.97041	22.95	*18.67*	18.12	0.96760	25.45	*20.76*	20.09	0.96471	27.95	*22.87*	22.06
0.97036	23.00	*18.71*	18.16	0.96755	25.50	*20.80*	20.13	0.96465	28.00	*22.91*	22.10
0.97030	23.05	*18.75*	18.20	0.96749	25.55	*20.84*	20.17	0.96459	28.05	*22.95*	22.14
0.97025	23.10	*18.79*	18.24	0.96744	25.60	*20.89*	20.21	0.96454	28.10	*23.00*	22.18
0.97019	23.15	*18.83*	18.28	0.96738	25.65	*20.93*	20.25	0.96448	28.15	*23.04*	22.22
0.97013	23.20	*18.88*	18.31	0.96733	25.70	*20.97*	20.29	0.96442	28.20	*23.08*	22.26
0.97007	23.25	*18.92*	18.35	0.96727	25.75	*21.01*	20.33	0.96436	28.25	*23.12*	22.30
0.97002	23.30	*18.96*	18.39	0.96722	25.80	*21.06*	20.37	0.96430	28.30	*23.17*	22.34
0.96996	23.35	*19.00*	18.43	0.96716	25.85	*21.10*	20.41	0.96424	28.35	*23.21*	22.38
0.96991	23.40	*19.04*	18.47	0.96710	25.90	*21.14*	20.44	0.96418	28.40	*23.25*	22.42
0.96985	23.45	*19.08*	18.51	0.96704	25.95	*21.18*	20.48	0.96412	28.45	*23.29*	22.46
0.96980	23.50	*19.13*	18.55	0.96699	26.00	*21.22*	20.52	0.96406	28.50	*23.33*	22.50
0.96974	23.55	*19.17*	18.59	0.96693	26.05	*21.26*	20.56	0.96400	28.55	*23.37*	22.54
0.96969	23.60	*19.21*	18.63	0.96687	26.10	*21.31*	20.60	0.96393	28.60	*23.42*	22.57
0.96963	23.65	*19.25*	18.67	0.96681	26.15	*21.35*	20.64	0.96387	28.65	*23.46*	22.61
0.96958	23.70	*19.29*	18.71	0.96675	26.20	*21.39*	20.68	0.96381	28.70	*23.51*	22.65
0.96952	23.75	*19.33*	18.75	0.96669	26.25	*21.43*	20.72	0.96375	28.75	*23.55*	22.69
0.96947	23.80	*19.38*	18.79	0.96664	26.30	*21.48*	20.76	0.96369	28.80	*23.59*	22.73
0.96941	23.85	*19.42*	18.83	0.96658	26.35	*21.52*	20.80	0.96363	28.85	*23.63*	22.77
0.96936	23.90	*19.46*	18.87	0.96653	26.40	*21.56*	20.84	0.96357	28.90	*23.67*	22.81
0.96930	23.95	*19.50*	18.91	0.96647	26.45	*21.60*	20.88	0.96351	28.95	*23.71*	22.85
0.96925	24.00	*19.55*	18.94	0.96641	26.50	*21.64*	20.92	0.96346	29.00	*23.76*	22.89
0.96919	24.05	*19.59*	18.98	0.96635	26.55	*21.68*	20.96	0.96340	29.05	*23.80*	22.93
0.96913	24.10	*19.63*	19.02	0.96630	26.60	*21.73*	21.00	0.96334	29.10	*23.84*	22.97
0.96907	24.15	*19.67*	19.06	0.96624	26.65	*21.77*	21.04	0.96328	29.15	*23.88*	23.01
0.96902	24.20	*19.71*	19.10	0.96618	26.70	*21.81*	21.08	0.96322	29.20	*23.93*	23.05

7

ALCOHOL TABLE.—Continued.

SPECIFIC GRAVITY 20° C./4°	ALCOHOL			SPECIFIC GRAVITY 20° C./4°	ALCOHOL			SPECIFIC GRAVITY 20° C./4°	ALCOHOL		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.96316	**29.25**	*23.97*	23.09	0.96005	**31.75**	*26.10*	25.06	0.95669	**34.25**	*28.26*	27.03
0.96310	**29.30**	*24.01*	23.13	0.95998	**31.80**	*26.15*	25.10	0.95662	**34.30**	*28.30*	27.07
0.96304	**29.35**	*24.05*	23.17	0.95992	**31.85**	*26.19*	25.14	0.95655	**34.35**	*28.34*	27.11
0.96297	**29.40**	*24.10*	23.21	0.95985	**31.90**	*26.23*	25.18	0.95648	**34.40**	*28.39*	27.15
0.96291	**29.45**	*24.14*	23.25	0.95979	**31.95**	*26.27*	25.22	0.95641	**34.45**	*28.43*	27.19
0.96285	**29.50**	*24.18*	23.29	0.95972	**32.00**	*26.32*	25.26	0.95634	**34.50**	*28.48*	27.23
0.96279	**29.55**	*24.22*	23.33	0.95965	**32.05**	*26.36*	25.30	0.95627	**34.55**	*28.52*	27.27
0.96273	**29.60**	*24.27*	23.36	0.95958	**32.10**	*26.41*	25.34	0.95619	**34.60**	*28.56*	27.31
0.96267	**29.65**	*24.31*	23.40	0.95952	**32.15**	*26.45*	25.38	0.95612	**34.65**	*28.60*	27.35
0.96261	**29.70**	*24.35*	23.44	0.95945	**32.20**	*26.49*	25.42	0.95605	**34.70**	*28.65*	27.39
0.96255	**29.75**	*24.39*	23.48	0.95939	**32.25**	*26.53*	25.46	0.95598	**34.75**	*28.69*	27.43
0.96248	**29.80**	*24.44*	23.52	0.95932	**32.30**	*26.58*	25.50	0.95591	**34.80**	*28.74*	27.47
0.96242	**29.85**	*24.48*	23.56	0.95926	**32.35**	*26.62*	25.54	0.95584	**34.85**	*28.78*	27.51
0.96236	**29.90**	*24.52*	23.60	0.95920	**32.40**	*26.66*	25.58	0.95577	**34.90**	*28.82*	27.55
0.96230	**29.95**	*24.56*	23.64	0.95913	**32.45**	*26.70*	25.61	0.95570	**34.95**	*28.86*	27.59
0.96224	**30.00**	*24.61*	23.68	0.95906	**32.50**	*26.75*	25.64	0.95563	**35.00**	*28.91*	27.63
0.96218	**30.05**	*24.65*	23.72	0.95900	**32.55**	*26.79*	25.68	0.95556	**35.05**	*28.95*	27.67
0.96211	**30.10**	*24.69*	23.76	0.95893	**32.60**	*26.83*	25.72	0.95549	**35.10**	*29.00*	27.71
0.96205	**30.15**	*24.73*	23.80	0.95887	**32.65**	*26.87*	25.76	0.95542	**35.15**	*29.04*	27.75
0.96199	**30.20**	*24.78*	23.84	0.95880	**32.70**	*26.92*	25.80	0.95535	**35.20**	*29.08*	27.78
0.96193	**30.25**	*24.82*	23.88	0.95873	**32.75**	*26.96*	25.84	0.95528	**35.25**	*29.12*	27.82
0.96187	**30.30**	*24.87*	23.92	0.95866	**32.80**	*27.01*	25.89	0.95521	**35.30**	*29.17*	27.86
0.96181	**30.35**	*24.91*	23.96	0.95859	**32.85**	*27.05*	25.93	0.95513	**35.35**	*29.21*	27.90
0.96175	**30.40**	*24.95*	24.00	0.95852	**32.90**	*27.09*	25.97	0.95506	**35.40**	*29.26*	27.94
0.96169	**30.45**	*24.99*	24.04	0.95846	**32.95**	*27.13*	26.01	0.95499	**35.45**	*29.30*	27.98
0.96163	**30.50**	*25.04*	24.08	0.95839	**33.00**	*27.18*	26.05	0.95492	**35.50**	*29.34*	28.02
0.96157	**30.55**	*25.08*	24.12	0.95833	**33.05**	*27.22*	26.09	0.95485	**35.55**	*29.38*	28.06
0.96150	**30.60**	*25.12*	24.15	0.95826	**33.10**	*27.27*	26.13	0.95478	**35.60**	*29.43*	28.10
0.96144	**30.65**	*25.16*	24.19	0.95819	**33.15**	*27.31*	26.17	0.95470	**35.65**	*29.47*	28.14
0.96138	**30.70**	*25.21*	24.23	0.95812	**33.20**	*27.35*	26.21	0.95463	**35.70**	*29.52*	28.18
0.96132	**30.75**	*25.25*	24.27	0.95806	**33.25**	*27.39*	26.25	0.95456	**35.75**	*29.56*	28.22
0.96125	**30.80**	*25.30*	24.31	0.95799	**33.30**	*27.44*	26.29	0.95449	**35.80**	*29.61*	28.26
0.96119	**30.85**	*25.34*	24.35	0.95792	**33.35**	*27.48*	26.33	0.95441	**35.85**	*29.65*	28.30
0.96112	**30.90**	*25.38*	24.39	0.95785	**33.40**	*27.52*	26.36	0.95434	**35.90**	*29.69*	28.34
0.96106	**30.95**	*25.42*	24.43	0.95778	**33.45**	*27.56*	26.40	0.95426	**35.95**	*29.73*	28.38
0.96100	**31.00**	*25.46*	24.47	0.95771	**33.50**	*27.61*	26.44	0.95419	**36.00**	*29.78*	28.42
0.96094	**31.05**	*25.50*	24.51	0.95764	**33.55**	*27.65*	26.48	0.95412	**36.05**	*29.82*	28.46
0.96088	**31.10**	*25.55*	24.55	0.95757	**33.60**	*27.70*	26.52	0.95405	**36.10**	*29.87*	28.49
0.96082	**31.15**	*25.59*	24.59	0.95751	**33.65**	*27.74*	26.56	0.95397	**36.15**	*29.91*	28.53
0.96075	**31.20**	*25.63*	24.63	0.95745	**33.70**	*27.78*	26.60	0.95390	**36.20**	*29.95*	28.57
0.96069	**31.25**	*25.67*	24.67	0.95738	**33.75**	*27.82*	26.64	0.95382	**36.25**	*29.99*	28.61
0.96062	**31.30**	*25.72*	24.71	0.95731	**33.80**	*27.87*	26.68	0.95375	**36.30**	*30.04*	28.65
0.96056	**31.35**	*25.76*	24.75	0.95724	**33.85**	*27.91*	26.72	0.95367	**36.35**	*30.09*	28.69
0.96049	**31.40**	*25.81*	24.79	0.95717	**33.90**	*27.96*	26.76	0.95360	**36.40**	*30.13*	28.73
0.96043	**31.45**	*25.85*	24.83	0.95710	**33.95**	*28.00*	26.80	0.95353	**36.45**	*30.17*	28.77
0.96036	**31.50**	*25.89*	24.86	0.95703	**34.00**	*28.04*	26.84	0.95346	**36.50**	*30.22*	28.81
0.96030	**31.55**	*25.93*	24.90	0.95696	**34.05**	*28.08*	26.88	0.95338	**36.55**	*30.26*	28.85
0.96024	**31.60**	*25.98*	24.94	0.95689	**34.10**	*28.13*	26.92	0.95331	**36.60**	*30.31*	28.89
0.96018	**31.65**	*26.02*	24.98	0.95682	**34.15**	*28.17*	26.96	0.95323	**36.65**	*30.35*	28.93
0.96011	**31.70**	*26.06*	25.02	0.95675	**34.20**	*28.22*	26.99	0.95315	**36.70**	*30.39*	28.97

7

ALCOHOL TABLE.—Continued.

SPECIFIC GRAVITY 20° C./4°	ALCOHOL			SPECIFIC GRAVITY 20° C./4°	ALCOHOL			SPECIFIC GRAVITY 20° C./4°	ALCOHOL		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.95308	36.75	*30.43*	29.01	0.94926	39.25	*32.63*	30.99	0.94519	41.75	*34.86*	32.96
0.95301	36.80	*30.48*	29.05	0.94918	39.30	*32.68*	31.02	0.94510	41.80	*34.91*	33.00
0.95294	36.85	*30.52*	29.09	0.94910	39.35	*32.72*	31.06	0.94502	41.85	*34.95*	33.04
0.95287	36.90	*30.57*	29.13	0.94901	39.40	*32.77*	31.10	0.94494	41.90	*35.00*	33.07
0.95279	36.95	*30.61*	29.17	0.94893	39.45	*32.81*	31.14	0.94486	41.95	*35.04*	33.11
0.95272	37.00	*30.66*	29.21	0.94885	39.50	*32.86*	31.18	0.94477	42.00	*35.09*	33.15
0.95264	37.05	*30.70*	29.25	0.94877	39.55	*32.90*	31.22	0.94469	42.05	*35.13*	33.19
0.95257	37.10	*30.74*	29.29	0.94869	39.60	*32.95*	31.26	0.94460	42.10	*35.18*	33.23
0.95249	37.15	*30.78*	29.33	0.94861	39.65	*32.99*	31.30	0.94452	42.15	*35.22*	33.27
0.95242	37.20	*30.83*	29.36	0.94853	39.70	*33.04*	31.34	0.94443	42.20	*35.27*	33.31
0.95234	37.25	*30.87*	29.40	0.94845	39.75	*33.08*	31.38	0.94435	42.25	*35.31*	33.35
0.95227	37.30	*30.92*	29.44	0.94837	39.80	*33.13*	31.42	0.94427	42.30	*35.36*	33.39
0.95219	37.35	*30.96*	29.48	0.94829	39.85	*33.17*	31.46	0.94419	42.35	*35.40*	33.43
0.95211	37.40	*31.01*	29.52	0.94821	39.90	*33.22*	31.50	0.94410	42.40	*35.45*	33.47
0.95203	37.45	*31.05*	29.56	0.94813	39.95	*33.26*	31.54	0.94402	42.45	*35.49*	33.51
0.95196	37.50	*31.09*	29.60	0.94805	40.00	*33.30*	31.57	0.94393	42.50	*35.54*	33.55
0.95188	37.55	*31.13*	29.64	0.94797	40.05	*33.34*	31.61	0.94385	42.55	*35.58*	33.59
0.95181	37.60	*31.18*	29.68	0.94789	40.10	*33.39*	31.65	0.94376	42.60	*35.63*	33.63
0.95173	37.65	*31.22*	29.72	0.94781	40.15	*33.43*	31.69	0.94368	42.65	*35.67*	33.67
0.95166	37.70	*31.27*	29.76	0.94773	40.20	*33.48*	31.73	0.94359	42.70	*35.72*	33.71
0.95158	37.75	*31.31*	29.80	0.94765	40.25	*33.52*	31.77	0.94351	42.75	*35.76*	33.75
0.95151	37.80	*31.36*	29.84	0.94757	40.30	*33.57*	31.81	0.94342	42.80	*35.81*	33.78
0.95143	37.85	*31.40*	29.88	0.94749	40.35	*33.61*	31.85	0.94334	42.85	*35.85*	33.82
0.95135	37.90	*31.45*	29.92	0.94741	40.40	*33.66*	31.89	0.94325	42.90	*35.90*	33.86
0.95127	37.95	*31.49*	29.96	0.94733	40.45	*33.70*	31.93	0.94317	42.95	*35.94*	33.90
0.95120	38.00	*31.53*	29.99	0.94725	40.50	*33.75*	31.97	0.94308	43.00	*35.99*	33.94
0.95112	38.05	*31.57*	30.03	0.94717	40.55	*33.79*	32.01	0.94300	43.05	*36.03*	33.98
0.95104	38.10	*31.62*	30.07	0.94708	40.60	*33.84*	32.05	0.94291	43.10	*36.08*	34.02
0.95096	38.15	*31.66*	30.11	0.94700	40.65	*33.88*	32.09	0.94283	43.15	*36.12*	34.06
0.95089	38.20	*31.71*	30.15	0.94692	40.70	*33.93*	32.13	0.94274	43.20	*36.17*	34.10
0.95081	38.25	*31.75*	30.19	0.94684	40.75	*33.97*	32.17	0.94265	43.25	*36.21*	34.14
0.95074	38.30	*31.80*	30.23	0.94676	40.80	*34.02*	32.20	0.94256	43.30	*36.26*	34.18
0.95066	38.35	*31.84*	30.27	0.94668	40.85	*34.06*	32.24	0.94248	43.35	*36.30*	34.22
0.95058	38.40	*31.89*	30.31	0.94659	40.90	*34.11*	32.28	0.94239	43.40	*36.35*	34.26
0.95050	38.45	*31.93*	30.35	0.94651	40.95	*34.15*	32.32	0.94231	43.45	*36.39*	34.30
0.95043	38.50	*31.97*	30.39	0.94643	41.00	*34.19*	32.36	0.94222	43.50	*36.44*	34.34
0.95035	38.55	*32.01*	30.43	0.94635	41.05	*34.23*	32.40	0.94214	43.55	*36.48*	34.38
0.95027	38.60	*32.06*	30.47	0.94627	41.10	*34.28*	32.44	0.94205	43.60	*36.53*	34.42
0.95019	38.65	*32.10*	30.51	0.94619	41.15	*34.32*	32.48	0.94197	43.65	*36.57*	34.46
0.95011	38.70	*32.15*	30.55	0.94610	41.20	*34.37*	32.52	0.94188	43.70	*36.62*	34.49
0.95003	38.75	*32.19*	30.59	0.94602	41.25	*34.41*	32.56	0.94179	43.75	*36.66*	34.53
0.94996	38.80	*32.24*	30.63	0.94594	41.30	*34.46*	32.60	0.94170	43.80	*36.71*	34.57
0.94988	38.85	*32.28*	30.67	0.94586	41.35	*34.50*	32.64	0.94161	43.85	*36.75*	34.61
0.94980	38.90	*32.33*	30.71	0.94577	41.40	*34.55*	32.68	0.94152	43.90	*36.80*	34.65
0.94972	38.95	*32.37*	30.75	0.94569	41.45	*34.59*	32.72	0.94144	43.95	*36.84*	34.69
0.94964	39.00	*32.42*	30.79	0.94560	41.50	*34.64*	32.76	0.94135	44.00	*36.89*	34.73
0.94956	39.05	*32.46*	30.83	0.94552	41.55	*34.68*	32.80	0.94126	44.05	*36.94*	34.77
0.94949	39.10	*32.51*	30.87	0.94544	41.60	*34.73*	32.84	0.94117	44.10	*36.99*	34.81
0.94941	39.15	*32.55*	30.91	0.94536	41.65	*34.77*	32.88	0.94108	44.15	*37.03*	34.85
0.94934	39.20	*32.59*	30.95	0.94527	41.70	*34.82*	32.92	0.94099	44.20	*37.08*	34.89

7

Alcohol Table.—Continued.

Specific gravity 20° C. / 4°	Alcohol: Per cent by volume at 20° C.	Alcohol: Per cent by weight	Alcohol: Grams per 100 cc.
0.94091	**44.25**	*37.12*	34.93
0.94082	**44.30**	*37.17*	34.97
0.94073	**44.35**	*37.21*	35.01
0.94064	**44.40**	*37.26*	35.05
0.94055	**44.45**	*37.30*	35.09
0.94046	**44.50**	*37.35*	35.13
0.94037	**44.55**	*37.39*	35.17
0.94028	**44.60**	*37.44*	35.20
0.94020	**44.65**	*37.48*	35.24
0.94011	**44.70**	*37.53*	35.28
0.94002	**44.75**	*37.57*	35.32
0.93993	**44.80**	*37.62*	35.36
0.93984	**44.85**	*37.66*	35.40
0.93975	**44.90**	*37.71*	35.44
0.93966	**44.95**	*37.75*	35.48
0.93957	**45.00**	*37.80*	35.52
0.93948	**45.05**	*37.85*	35.56
0.93939	**45.10**	*37.90*	35.60
0.93931	**45.15**	*37.94*	35.64
0.93922	**45.20**	*37.99*	35.68
0.93912	**45.25**	*38.03*	35.72
0.93903	**45.30**	*38.08*	35.76
0.93894	**45.35**	*38.12*	35.80
0.93885	**45.40**	*38.17*	35.84
0.93876	**45.45**	*38.21*	35.88
0.93867	**45.50**	*38.26*	35.92
0.93858	**45.55**	*38.30*	35.96
0.93849	**45.60**	*38.35*	35.99
0.93840	**45.65**	*38.39*	36.03
0.93831	**45.70**	*38.44*	36.07
0.93822	**45.75**	*38.49*	36.11
0.93813	**45.80**	*38.54*	36.15
0.93803	**45.85**	*38.58*	36.19
0.93794	**45.90**	*38.63*	36.23
0.93785	**45.95**	*38.67*	36.27
0.93776	**46.00**	*38.72*	36.31
0.93767	**46.05**	*38.76*	36.35
0.93758	**46.10**	*38.81*	36.39
0.93749	**46.15**	*38.85*	36.43
0.93740	**46.20**	*38.90*	36.47
0.93730	**46.25**	*38.95*	36.51
0.93721	**46.30**	*39.00*	36.55
0.93712	**46.35**	*39.04*	36.59
0.93703	**46.40**	*39.09*	36.63
0.93693	**46.45**	*39.13*	36.67
0.93684	**46.50**	*39.18*	36.70
0.93675	**46.55**	*39.22*	36.74
0.93666	**46.60**	*39.27*	36.78
0.93656	**46.65**	*39.31*	36.82
0.93647	**46.70**	*39.36*	36.86
0.93638	**46.75**	*39.41*	36.90
0.93629	**46.80**	*39.46*	36.94
0.93619	**46.85**	*39.50*	36.98
0.93610	**46.90**	*39.55*	37.02
0.93600	**46.95**	*39.59*	37.06
0.93591	**47.00**	*39.64*	37.10
0.93582	**47.05**	*39.68*	37.14
0.93573	**47.10**	*39.73*	37.18
0.93563	**47.15**	*39.77*	37.22
0.93554	**47.20**	*39.82*	37.26
0.93545	**47.25**	*39.87*	37.30
0.93536	**47.30**	*39.92*	37.34
0.93526	**47.35**	*39.96*	37.38
0.93517	**47.40**	*40.01*	37.42
0.93507	**47.45**	*40.05*	37.46
0.93498	**47.50**	*40.10*	37.49
0.93488	**47.55**	*40.14*	37.53
0.93479	**47.60**	*40.19*	37.57
0.93470	**47.65**	*40.24*	37.61
0.93461	**47.70**	*40.29*	37.65
0.93451	**47.75**	*40.33*	37.69
0.93442	**47.80**	*40.38*	37.73
0.93432	**47.85**	*40.42*	37.77
0.93423	**47.90**	*40.47*	37.81
0.93413	**47.95**	*40.51*	37.85
0.93404	**48.00**	*40.56*	37.89
0.93394	**48.05**	*40.61*	37.93
0.93385	**48.10**	*40.66*	37.97
0.93375	**48.15**	*40.70*	38.01
0.93366	**48.20**	*40.75*	38.05
0.93356	**48.25**	*40.79*	38.09
0.93347	**48.30**	*40.84*	38.13
0.93337	**48.35**	*40.89*	38.17
0.93328	**48.40**	*40.94*	38.21
0.93318	**48.45**	*40.98*	38.25
0.93308	**48.50**	*41.03*	38.29
0.93298	**48.55**	*41.07*	38.33
0.93289	**48.60**	*41.12*	38.36
0.93279	**48.65**	*41.16*	38.40
0.93270	**48.70**	*41.21*	38.44
0.93260	**48.75**	*41.26*	38.48
0.93251	**48.80**	*41.31*	38.52
0.93241	**48.85**	*41.35*	38.56
0.93232	**48.90**	*41.40*	38.60
0.93222	**48.95**	*41.44*	38.64
0.93213	**49.00**	*41.49*	38.68
0.93203	**49.05**	*41.54*	38.72
0.93194	**49.10**	*41.59*	38.76
0.93184	**49.15**	*41.63*	38.80
0.93174	**49.20**	*41.68*	38.83
0.93164	**49.25**	*41.72*	38.87
0.93155	**49.30**	*41.77*	38.91
0.93145	**49.35**	*41.82*	38.95
0.93136	**49.40**	*41.87*	38.99
0.93126	**49.45**	*41.91*	39.03
0.93116	**49.50**	*41.96*	39.07
0.93106	**49.55**	*42.01*	39.11
0.93096	**49.60**	*42.06*	39.15
0.93086	**49.65**	*42.10*	39.19
0.93076	**49.70**	*42.15*	39.23
0.93066	**49.75**	*42.19*	39.27
0.93056	**49.80**	*42.24*	39.31
0.93046	**49.85**	*42.29*	39.35
0.93036	**49.90**	*42.34*	39.39
0.93026	**49.95**	*42.38*	39.43
0.93017	**50.00**	*42.43*	39.47
0.93007	**50.05**	*42.47*	39.51
0.92997	**50.10**	*42.52*	39.55
0.92987	**50.15**	*42.57*	39.59
0.92977	**50.20**	*42.62*	39.63
0.92967	**50.25**	*42.66*	39.67
0.92957	**50.30**	*42.71*	39.70
0.92947	**50.35**	*42.76*	39.74
0.92938	**50.40**	*42.81*	39.78
0.92928	**50.45**	*42.85*	39.82
0.92918	**50.50**	*42.90*	39.86
0.92908	**50.55**	*42.94*	39.90
0.92898	**50.60**	*42.99*	39.94
0.92888	**50.65**	*43.04*	39.98
0.92879	**50.70**	*43.09*	40.02
0.92869	**50.75**	*43.13*	40.06
0.92859	**50.80**	*43.18*	40.10
0.92849	**50.85**	*43.23*	40.14
0.92839	**50.90**	*43.28*	40.18
0.92829	**50.95**	*43.32*	40.22
0.92818	**51.00**	*43.37*	40.26
0.92808	**51.05**	*43.42*	40.30
0.92798	**51.10**	*43.47*	40.34
0.92788	**51.15**	*43.51*	40.38
0.92778	**51.20**	*43.56*	40.42
0.92768	**51.25**	*43.60*	40.46
0.92759	**51.30**	*43.65*	40.49
0.92749	**51.35**	*43.70*	40.53
0.92739	**51.40**	43.75	40.57
0.92729	**51.45**	*43.79*	40.61
0.92719	**51.50**	*43.84*	40.65
0.92709	**51.55**	*43.89*	40.69
0.92699	**51.60**	*43.94*	40.73
0.92689	**51.65**	*43.98*	40.77
0.92678	**51.70**	*44.03*	40.81

7

Alcohol Table.—Continued.

Specific gravity 20° C. / 4°	Alcohol			Specific gravity 20° C. / 4°	Alcohol			Specific gravity 20° C. / 4°	Alcohol		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.92668	51.75	44.08	40.85	0.92157	54.25	46.46	42.82	0.91629	56.75	48.89	44.80
0.92658	51.80	44.13	40.89	0.92147	54.30	46.51	42.86	0.91618	56.80	48.94	44.83
0.92648	51.85	44.17	40.93	0.92137	54.35	46.56	42.90	0.91608	56.85	48.98	44.87
0.92637	51.90	44.22	40.97	0.92126	54.40	46.61	42.94	0.91597	56.90	49.03	44.91
0.92627	51.95	44.26	41.01	0.92116	54.45	46.66	42.98	0.91586	56.95	49.08	44.95
0.92617	52.00	44.31	41.05	0.92105	54.50	46.71	43.02	0.91575	57.00	49.13	44.99
0.92607	52.05	44.36	41.09	0.92095	54.55	46.75	43.06	0.91565	57.05	49.18	45.03
0.92597	52.10	44.41	41.13	0.92084	54.60	46.80	43.10	0.91554	57.10	49.23	45.07
0.92587	52.15	44.46	41.17	0.92074	54.65	46.85	43.14	0.91543	57.15	49.28	45.11
0.92577	52.20	44.51	41.20	0.92063	54.70	46.90	43.18	0.91532	57.20	49.33	45.15
0.92567	52.25	44.55	41.24	0.92053	54.75	46.94	43.22	0.91521	57.25	49.38	45.19
0.92557	52.30	44.60	41.28	0.92042	54.80	46.99	43.26	0.91510	57.30	49.43	45.23
0.92547	52.35	44.65	41.32	0.92032	54.85	47.04	43.30	0.91500	57.35	49.47	45.27
0.92537	52.40	44.70	41.36	0.92021	54.90	47.09	43.34	0.91489	57.40	49.52	45.31
0.92527	52.45	44.74	41.40	0.92011	54.95	47.14	43.38	0.91478	57.45	49.57	45.35
0.92516	52.50	44.79	41.44	0.92000	55.00	47.19	43.42	0.91467	57.50	49.62	45.39
0.92506	52.55	44.84	41.48	0.91990	55.05	47.24	43.46	0.91457	57.55	49.67	45.43
0.92496	52.60	44.89	41.52	0.91979	55.10	47.29	43.49	0.91446	59.60	49.72	45.47
0.92486	52.65	44.93	41.56	0.91969	55.15	47.33	43.53	0.91435	57.65	49.77	45.51
0.92476	52.70	44.98	41.60	0.91958	55.20	47.38	43.57	0.91424	57.70	49.82	45.55
0.92466	52.75	45.03	41.64	0.91948	55.25	47.43	43.61	0.91414	57.75	49.87	45.59
0.92455	52.80	45.08	41.68	0.91937	55.30	47.48	43.65	0.91403	57.80	49.92	45.63
0.92445	52.85	45.12	41.72	0.91927	55.35	47.53	43.69	0.91392	57.85	49.96	45.67
0.92434	52.90	45.17	41.76	0.91916	55.40	47.58	43.73	0.91381	57.90	50.01	45.70
0.92424	52.95	45.22	41.80	0.91906	55.45	47.62	43.77	0.91370	57.95	50.06	45.74
0.92414	53.00	45.27	41.83	0.91895	55.50	47.67	43.81	0.91359	58.00	50.11	45.78
0.92404	53.05	45.31	41.87	0.91885	55.55	47.72	43.85	0.91348	58.05	50.16	45.82
0.92394	53.10	45.36	41.91	0.91874	55.60	47.77	43.89	0.91337	58.10	50.21	45.86
0.92384	53.15	45.41	41.95	0.91864	55.65	47.82	43.93	0.91326	58.15	50.26	45.90
0.92373	53.20	45.46	41.99	0.91853	55.70	47.87	43.97	0.91315	58.20	50.31	45.94
0.92363	53.25	45.51	42.03	0.91842	55.75	47.91	44.01	0.91304	58.25	50.36	45.98
0.92353	53.30	45.56	42.07	0.91831	55.80	47.96	44.04	0.91293	58.30	50.41	46.02
0.92343	53.35	45.60	42.11	0.91821	55.85	48.01	44.08	0.91282	58.35	50.46	46.06
0.92332	53.40	45.65	41.15	0.91810	55.90	48.06	44.12	0.91271	58.40	50.51	46.10
0.92322	53.45	45.70	42.19	0.91800	55.95	48.11	44.16	0.91261	58.45	50.55	46.14
0.92312	53.50	45.75	42.23	0.91789	56.00	48.16	44.20	0.91250	58.50	50.60	46.17
0.92302	53.55	45.79	42.27	0.91779	56.05	48.20	44.24	0.91239	58.55	50.65	46.21
0.92291	53.60	45.84	42.31	0.91768	56.10	48.25	44.28	0.91228	58.60	50.70	46.25
0.92281	53.65	45.89	42.35	0.91758	56.15	48.30	44.32	0.91217	58.65	50.75	46.29
0.92271	53.70	45.94	42.39	0.91747	56.20	48.35	44.36	0.91206	58.70	50.80	46.33
0.92261	53.75	45.98	42.43	0.91736	56.25	48.40	44.40	0.91194	58.75	50.85	46.37
0.92250	53.80	46.03	42.47	0.91725	56.30	48.45	44.44	0.91183	58.80	50.90	46.41
0.92240	53.85	46.08	42.51	0.91715	56.35	48.50	44.48	0.91171	58.85	50.95	46.45
0.92230	53.90	46.13	42.55	0.91704	56.40	48.55	44.52	0.91160	58.90	51.00	46.49
0.92220	53.95	46.18	42.59	0.91694	56.45	48.59	44.56	0.91149	58.95	51.05	46.53
0.92209	54.00	46.23	42.62	0.91683	56.50	48.64	44.60	0.91138	59.00	51.10	46.57
0.92199	54.05	46.27	42.66	0.91672	56.55	48.69	44.64	0.91127	59.05	51.15	46.61
0.92188	54.10	46.32	42.70	0.91661	56.60	48.74	44.68	0.91116	59.10	51.20	46.65
0.92178	54.15	46.36	42.74	0.91650	56.65	48.79	44.72	0.91104	59.15	51.25	46.69
0.92167	54.20	46.41	42.78	0.91639	56.70	48.84	44.76	0.91093	59.20	51.30	46.73

7

Alcohol Table.—Continued.

SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.91082	**59.25**	*51.35*	46.77
0.91071	**59.30**	*51.40*	46.81
0.91060	**59.35**	*51.45*	46.85
0.91049	**59.40**	*51.50*	46.89
0.91038	**59.45**	*51.55*	46.93
0.91027	**59.50**	*51.60*	46.97
0.91016	**59.55**	*51.65*	47.01
0.91005	**59.60**	*51.70*	47.05
0.90993	**59.65**	*51.74*	47.09
0.90982	**59.70**	*51.79*	47.12
0.90971	**59.75**	*51.84*	47.16
0.90960	**59.80**	*51.89*	47.20
0.90949	**59.85**	*51.94*	47.24
0.90938	**59.90**	*51.99*	47.28
0.90926	**59.95**	*52.04*	47.32
0.90915	**60.00**	*52.09*	47.36
0.90904	**60.05**	*52.14*	47.40
0.90893	**60.10**	*52.19*	47.44
0.90882	**60.15**	*52.24*	47.48
0.90871	**60.20**	*52.29*	47.52
0.90859	**60.25**	*52.34*	47.56
0.90848	**60.30**	*52.39*	47.60
0.90837	**60.35**	*52.44*	47.64
0.90826	**60.40**	*52.49*	47.68
0.90814	**60.45**	*52.54*	47.72
0.90803	**60.50**	*52.59*	47.76
0.90792	**60.55**	*52.64*	47.80
0.90781	**60.60**	*52.69*	47.84
0.90769	**60.65**	*52.74*	47.88
0.90758	**60.70**	*52.79*	47.91
0.90747	**60.75**	*52.84*	47.95
0.90736	**60.80**	*52.89*	47.99
0.90724	**60.85**	*52.94*	48.03
0.90713	**60.90**	*52.99*	48.07
0.90701	**60.95**	*53.04*	48.11
0.90690	**61.00**	*53.09*	48.15
0.90678	**61.05**	*53.14*	48.19
0.90667	**61.10**	*53.19*	48.23
0.90656	**61.15**	*53.24*	48.27
0.90645	**61.20**	*53.29*	48.31
0.90633	**61.25**	*53.34*	48.35
0.90622	**61.30**	*53.39*	48.39
0.90610	**61.35**	*53.44*	48.43
0.90599	**61.40**	*53.49*	48.47
0.90588	**61.45**	*53.55*	48.51
0.90577	**61.50**	*53.60*	48.55
0.90565	**61.55**	*53.65*	48.59
0.90554	**61.60**	*53.70*	48.62
0.90543	**61.65**	*53.75*	48.66
0.90532	**61.70**	*53.80*	48.70
0.90520	**61.75**	*53.85*	48.74
0.90509	**61.80**	*53.90*	48.78
0.90497	**61.85**	*53.95*	48.82
0.90486	**61.90**	*54.00*	48.86
0.90474	**61.95**	*54.05*	48.90
0.90463	**62.00**	*54.10*	48.94
0.90451	**62.05**	*54.15*	48.98
0.90440	**62.10**	*54.20*	49.02
0.90428	**62.15**	*54.25*	49.06
0.90417	**62.20**	*54.30*	49.10
0.90406	**62.25**	*54.35*	49.14
0.90395	**62.30**	*54.40*	49.18
0.90383	**62.35**	*54.45*	49.22
0.90372	**62.40**	*54.50*	49.25
0.90360	**62.45**	*54.55*	49.29
0.90349	**62.50**	*54.60*	49.33
0.90337	**62.55**	*54.66*	49.37
0.90326	**62.60**	*54.71*	49.41
0.90314	**62.65**	*54.76*	49.45
0.90302	**62.70**	*54.81*	49.49
0.90290	**62.75**	*54.86*	49.53
0.90279	**62.80**	*54.91*	49.57
0.90267	**62.85**	*54.96*	49.61
0.90256	**62.90**	*55.01*	49.65
0.90244	**62.95**	*55.06*	49.69
0.90233	**63.00**	*55.11*	49.73
0.90221	**63.05**	*55.16*	49.77
0.90210	**63.10**	*55.21*	49.81
0.90198	**63.15**	*55.26*	49.85
0.90187	**63.20**	*55.31*	49.89
0.90175	**63.25**	*55.37*	49.93
0.90163	**63.30**	*55.42*	49.97
0.90151	**63.35**	*55.47*	50.01
0.90140	**63.40**	*55.52*	50.04
0.90128	**63.45**	*55.57*	50.08
0.90117	**63.50**	*55.62*	50.12
0.90105	**63.55**	*55.67*	50.16
0.90094	**63.60**	*55.72*	50.20
0.90082	**63.65**	*55.77*	50.24
0.90070	**63.70**	*55.82*	50.28
0.90059	**63.75**	*55.88*	50.32
0.90048	**63.80**	*55.93*	50.36
0.90036	**63.85**	*55.98*	50.40
0.90025	**63.90**	*56.03*	50.44
0.90013	**63.95**	*56.08*	50.48
0.90001	**64.00**	*56.13*	50.52
0.89989	**64.05**	*56.18*	50.56
0.89978	**64.10**	*56.23*	50.60
0.89966	**64.15**	*56.29*	50.64
0.89954	**64.20**	*56.34*	50.68
0.89942	**64.25**	*56.39*	50.72
0.89930	**64.30**	*56.44*	50.76
0.89918	**64.35**	*56.49*	50.80
0.89907	**64.40**	*56.54*	50.83
0.89895	**64.45**	*56.59*	50.87
0.89884	**64.50**	*56.64*	50.91
0.89872	**64.55**	*56.70*	50.95
0.89861	**64.60**	*56.75*	50.99
0.89849	**64.65**	*56.80*	51.03
0.89837	**64.70**	*56.85*	51.07
0.89825	**64.75**	*56.90*	51.11
0.89814	**64.80**	*56.95*	51.15
0.89802	**64.85**	*57.00*	51.19
0.89791	**64.90**	*57.05*	51.23
0.89779	**64.95**	*57.11*	51.27
0.89767	**65.00**	*57.16*	51.31
0.89755	**65.05**	*57.21*	51.35
0.89744	**65.10**	*57.26*	51.39
0.89732	**65.15**	*57.31*	51.43
0.89720	**65.20**	*57.36*	51.47
0.89708	**65.25**	*57.41*	51.51
0.89696	**65.30**	*57.46*	51.55
0.89684	**65.35**	*57.52*	51.59
0.89672	**65.40**	*57.57*	51.63
0.89660	**65.45**	*57.62*	51.67
0.89649	**65.50**	*57.67*	51.71
0.89637	**65.55**	*57.72*	51.75
0.89626	**65.60**	*57.77*	51.78
0.89614	**65.65**	*57.83*	51.82
0.89602	**65.70**	*57.88*	51.86
0.89590	**65.75**	*57.93*	51.90
0.89578	**65.80**	*57.98*	51.94
0.89566	**65.85**	*58.04*	51.98
0.89554	**65.90**	*58.09*	52.02
0.89542	**65.95**	*58.14*	52.06
0.89531	**66.00**	*58.19*	52.10
0.89519	**66.05**	*58.24*	52.14
0.89507	**66.10**	*58.29*	52.18
0.89495	**66.15**	*58.35*	52.22
0.89483	**66.20**	*58.40*	52.26
0.89471	**66.25**	*58.45*	52.30
0.89459	**66.30**	*58.50*	52.33
0.89447	**66.35**	*58.55*	52.37
0.89435	**66.40**	*58.60*	52.41
0.89423	**66.45**	*58.66*	52.45
0.89411	**66.50**	*58.71*	52.49
0.89399	**66.55**	*58.76*	52.53
0.89387	**66.60**	*58.81*	52.57
0.89375	**66.65**	*58.87*	52.61
0.89363	**66.70**	*58.92*	52.65

7

ALCOHOL TABLE.—Continued.

SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL			SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL			SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.89351	66.75	58.97	52.69	0.88744	69.25	61.60	54.66	0.88120	71.75	64.27	56.64
0.89339	66.80	59.02	52.73	0.88732	69.30	61.65	54.70	0.88107	71.80	64.32	56.68
0.89327	66.85	59.07	52.77	0.88720	69.35	61.70	54.74	0.88094	71.85	64.38	56.72
0.89315	66.90	59.12	52.81	0.88707	69.40	61.75	54.78	0.88081	71.90	64.43	56.75
0.89303	66.95	59.18	52.85	0.88695	69.45	61.81	54.82	0.88069	71.95	64.49	56.79
0.89291	67.00	59.23	52.89	0.88682	69.50	61.86	54.86	0.88056	72.00	64.54	56.83
0.89279	67.05	59.28	52.93	0.88670	69.55	61.92	54.90	0.88044	72.05	64.60	56.87
0.89267	67.10	59.33	52.97	0.88658	69.60	61.97	54.94	0.88031	72.10	64.65	56.91
0.89255	67.15	59.39	53.01	0.88646	69.65	62.02	54.98	0.88018	72.15	64.71	56.95
0.89243	67.20	59.44	53.04	0.88633	69.70	62.07	55.02	0.88005	72.20	64.76	56.99
0.89231	67.25	59.49	53.08	0.88621	69.75	62.13	55.06	0.87993	72.25	64.82	57.03
0.89219	67.30	59.54	53.12	0.88608	69.80	62.18	55.10	0.87980	72.30	64.87	57.07
0.89207	67.35	59.60	53.16	0.88596	69.85	62.24	55.14	0.87967	72.35	64.93	57.11
0.89195	67.40	59.65	53.20	0.88583	69.90	62.29	55.18	0.87954	72.40	64.98	57.15
0.89183	67.45	59.70	53.24	0.88571	69.95	62.34	55.22	0.87942	72.45	65.03	57.19
0.89171	67.50	59.75	53.28	0.88558	70.00	62.39	55.25	0.87929	72.50	65.08	57.23
0.89159	67.55	59.81	53.32	0.88546	70.05	62.45	55.29	0.87916	72.55	65.14	57.27
0.89147	67.60	59.86	53.36	0.99533	70.10	62.50	55.33	0.87903	72.60	65.19	57.31
0.89135	67.65	59.91	53.40	0.88521	70.15	62.56	55.37	0.87891	72.65	65.25	57.35
0.89122	67.70	59.96	53.44	0.88508	70.20	62.61	55.41	0.87878	72.70	65.30	57.38
0.89110	67.75	60.02	53.48	0.88496	70.25	62.66	55.45	0.87865	72.75	65.36	57.42
0.89098	67.80	60.07	53.52	0.88484	70.30	62.71	55.49	0.87852	72.80	65.41	57.46
0.89086	67.85	60.12	53.56	0.88472	70.35	62.77	55.53	0.87839	72.85	65.47	57.50
0.89074	67.90	60.17	53.60	0.88459	70.40	62.82	55.57	0.87826	72.90	65.52	57.54
0.89062	67.95	60.23	53.64	0.88447	70.45	62.87	55.61	0.87813	72.95	65.58	57.58
0.89050	68.00	60.28	53.68	0.88434	70.50	62.92	55.65	0.87800	73.00	65.63	57.62
0.89038	68.05	60.33	53.72	0.88422	70.55	62.98	55.69	0.87788	73.05	65.69	57.66
0.89026	68.10	60.38	53.75	0.88409	70.60	63.03	55.73	0.87775	73.10	65.74	57.70
0.89014	68.15	60.44	53.79	0.88397	70.65	63.09	55.77	0.87762	73.15	65.80	57.74
0.89001	68.20	60.49	53.83	0.88384	70.70	63.14	55.81	0.87749	73.20	65.85	57.78
0.88989	68.25	60.54	53.87	0.88372	70.75	63.20	55.85	0.87737	73.25	65.91	57.82
0.88977	68.30	60.59	53.91	0.88359	70.80	63.25	55.89	0.87724	73.30	65.96	57.86
0.88965	68.35	60.65	53.95	0.88347	70.85	63.31	55.93	0.87711	73.35	66.02	57.90
0.88952	68.40	60.70	53.99	0.88334	70.90	63.36	55.97	0.87698	73.40	66.07	57.94
0.88940	68.45	60.75	54.03	0.88322	70.95	63.41	56.01	0.87685	73.45	66.13	57.98
0.88928	68.50	60.80	54.07	0.88309	71.00	63.46	56.04	0.87672	73.50	66.18	58.02
0.88916	68.55	60.86	54.11	0.88297	71.05	63.52	56.08	0.87659	73.55	66.23	58.06
0.88904	68.60	60.91	54.15	0.88284	71.10	63.57	56.12	0.87646	73.60	66.28	58.10
0.88892	68.65	60.96	54.19	0.88272	71.15	63.63	56.16	0.87633	73.65	66.34	58.14
0.88879	68.70	61.01	54.23	0.88259	71.20	63.68	56.20	0.87620	73.70	66.39	58.17
0.88867	68.75	61.07	54.27	0.88246	71.25	63.74	56.24	0.87607	73.75	66.45	58.21
0.88854	68.80	61.12	54.31	0.88233	71.30	63.79	56.28	0.87594	73.80	66.50	58.25
0.88842	68.85	61.17	54.35	0.88221	71.35	63.84	56.32	0.87581	73.85	66.56	58.29
0.88830	68.90	61.22	54.39	0.88208	71.40	63.89	56.36	0.87568	73.90	66.61	58.33
0.88818	68.95	61.28	54.43	0.88196	71.45	63.95	56.40	0.87555	73.95	66.67	58.37
0.88805	69.00	61.33	54.47	0.88183	71.50	64.00	56.44	0.87542	74.00	66.72	58.41
0.88793	69.05	61.39	54.51	0.88171	71.55	64.06	56.48	0.87529	74.05	66.78	58.45
0.88781	69.10	61.44	54.54	0.88158	71.60	64.11	56.52	0.87516	74.10	66.83	58.49
0.88769	69.15	61.49	54.58	0.88145	71.65	64.17	56.56	0.87504	74.15	66.89	58.53
0.88756	69.20	61.54	54.62	0.88132	71.70	64.22	56.60	0.87491	74.20	66.94	58.57

Alcohol Table.—Continued.

SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL			SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL			SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.87478	**74.25**	*67.00*	58.61	0.86818	**76.75**	*69.78*	60.58	0.86137	**79.25**	*72.63*	62.56
0.87465	**74.30**	*67.05*	58.65	0.86805	**76.80**	*69.84*	60.62	0.86124	**79.30**	*72.68*	62.60
0.87452	**74.35**	*67.11*	58.69	0.86791	**76.85**	*69.90*	60.66	0.86110	**79.35**	*72.74*	62.64
0.87439	**74.40**	*67.16*	58.73	0.86778	**76.90**	*69.95*	60.70	0.86096	**79.40**	*72.80*	62.67
0.87426	**74.45**	*67.22*	58.77	0.86764	**76.95**	*70.01*	60.74	0.86082	**79.45**	*72.86*	62.71
0.87413	**74.50**	*67.27*	58.81	0.86751	**77.00**	*70.06*	60.78	0.86069	**79.50**	*72.91*	62.75
0.87400	**74.55**	*67.33*	58.85	0.86737	**77.05**	*70.12*	60.82	0.86055	**79.55**	*72.97*	62.79
0.87387	**74.60**	*67.38*	58.89	0.86724	**77.10**	*70.18*	60.86	0.86041	**79.60**	*73.03*	62.83
0.87373	**74.65**	*67.44*	58.93	0.86710	**77.15**	*70.24*	60.90	0.86027	**79.65**	*73.09*	62.87
0.87360	**74.70**	*67.49*	58.97	0.86697	**77.20**	*70.29*	60.94	0.86013	**79.70**	*73.14*	62.91
0.87347	**74.75**	*67.55*	59.01	0.86684	**77.25**	*70.35*	60.98	0.85999	**79.75**	*73.20*	62.95
0.87334	**74.80**	*67.61*	59.04	0.86671	**77.30**	*70.40*	61.02	0.85984	**79.80**	*73.26*	62.99
0.87321	**74.85**	*67.67*	59.08	0.86657	**77.35**	*70.46*	61.06	0.85970	**79.85**	*73.32*	63.03
0.87308	**74.90**	*67.72*	59.12	0.86644	**77.40**	*70.51*	61.10	0.85956	**79.90**	*73.37*	63.07
0.87295	**74.95**	*67.78*	59.16	0.86630	**77.45**	*70.57*	61.14	0.85942	**79.95**	*73.43*	63.11
0.87282	**75.00**	*67.83*	59.20	0.86617	**77.50**	*70.63*	61.18	0.85928	**80.00**	*73.49*	63.15
0.87269	**75.05**	*67.89*	59.24	0.86603	**77.55**	*70.69*	61.22	0.85914	**80.05**	*73.55*	63.19
0.87256	**75.10**	*67.94*	59.28	0.86589	**77.60**	*70.74*	61.25	0.85901	**80.10**	*73.60*	63.23
0.87243	**75.15**	*68.00*	59.32	0.86575	**77.65**	*70.80*	61.29	0.85887	**80.15**	*73.66*	63.27
0.87230	**75.20**	*68.05*	59.36	0.86562	**77.70**	*70.85*	61.33	0.85873	**80.20**	*73.72*	63.30
0.87217	**75.25**	*68.11*	59.40	0.86548	**77.75**	*70.91*	61.37	0.85859	**80.25**	*73.78*	63.34
0.87204	**75.30**	*68.16*	59.44	0.86535	**77.80**	*70.97*	61.41	0.85846	**80.30**	*73.83*	63.38
0.87190	**75.35**	*68.22*	59.48	0.86521	**77.85**	*71.03*	61.45	0.85832	**80.35**	*73.89*	63.42
0.87177	**75.40**	*68.27*	59.52	0.86508	**77.90**	*71.08*	61.49	0.85818	**80.40**	*73.95*	63.46
0.87164	**75.45**	*68.33*	59.56	0.86494	**77.95**	*71.14*	61.53	0.85804	**80.45**	*74.01*	63.50
0.87151	**75.50**	*68.38*	59.60	0.86480	**78.00**	*71.19*	61.57	0.85789	**80.50**	*74.06*	63.54
0.87138	**75.55**	*68.44*	59.64	0.86466	**78.05**	*71.25*	61.61	0.85775	**80.55**	*74.12*	63.58
0.87125	**75.60**	*68.49*	59.67	0.86453	**78.10**	*71.31*	61.65	0.85761	**80.60**	*74.18*	63.62
0.87111	**75.65**	*68.55*	59.71	0.86439	**78.15**	*71.37*	61.69	0.85747	**80.65**	*74.24*	63.66
0.87098	**75.70**	*68.60*	59.75	0.86426	**78.20**	*71.42*	61.73	0.85733	**80.70**	*74.30*	63.70
0.87084	**75.75**	*68.66*	59.79	0.86412	**78.25**	*71.48*	61.77	0.85719	**80.75**	*74.36*	63.74
0.87071	**75.80**	*68.72*	59.83	0.86399	**78.30**	*71.54*	61.81	0.85705	**80.80**	*74.42*	63.78
0.87058	**75.85**	*68.78*	59.87	0.86385	**78.35**	*71.60*	61.85	0.85691	**80.85**	*74.48*	63.82
0.87045	**75.90**	*68.83*	59.91	0.86371	**78.40**	*71.65*	61.88	0.85677	**80.90**	*74.53*	63.86
0.87032	**75.95**	*68.89*	59.95	0.86357	**78.45**	*71.71*	61.92	0.85663	**80.95**	*74.59*	63.90
0.87019	**76.00**	*68.94*	59.99	0.86344	**78.50**	*71.76*	61.96	0.85648	**81.00**	*74.65*	63.94
0.87005	**76.05**	*69.00*	60.03	0.86330	**78.55**	*71.82*	62.00	0.85634	**81.05**	*74.71*	63.98
0.86992	**76.10**	*69.05*	60.07	0.86316	**78.60**	*71.88*	62.04	0.85620	**81.10**	*74.77*	64.02
0.86979	**76.15**	*69.11*	60.11	0.86302	**78.65**	*71.94*	62.08	0.85606	**81.15**	*74.83*	64.06
0.86966	**76.20**	*69.16*	60.15	0.86289	**78.70**	*71.99*	62.12	0.85592	**81.20**	*74.88*	64.09
0.86952	**76.25**	*69.22*	60.19	0.86275	**78.75**	*72.05*	62.16	0.85578	**81.25**	*74.94*	64.13
0.86939	**76.30**	*69.27*	60.23	0.86261	**78.80**	*72.11*	62.20	0.85564	**81.30**	*75.00*	64.17
0.86925	**76.35**	*69.33*	60.27	0.86247	**78.85**	*72.17*	62.24	0.85550	**81.35**	*75.06*	64.21
0.86912	**76.40**	*69.39*	60.31	0.86234	**78.90**	*72.22*	62.28	0.85536	**81.40**	*75.12*	64.25
0.86898	**76.45**	*69.45*	60.35	0.86220	**78.95**	*72.28*	62.32	0.85522	**81.45**	*75.18*	64.29
0.86885	**76.50**	*69.50*	60.39	0.86206	**79.00**	*72.34*	62.36	0.85507	**81.50**	*75.24*	64.33
0.86872	**76.55**	*69.56*	60.43	0.86192	**79.05**	*72.40*	62.40	0.85493	**81.55**	*75.30*	64.37
0.86859	**76.60**	*69.61*	60.47	0.86179	**79.10**	*72.45*	62.44	0.85478	**81.60**	*75.35*	64.41
0.86845	**76.65**	*69.67*	60.51	0.86165	**79.15**	*72.51*	62.48	0.85464	**81.65**	*75.41*	64.45
0.86832	**76.70**	*69.72*	60.54	0.86151	**79.20**	*72.57*	62.52	0.85450	**81.70**	*75.47*	64.49

7

Alcohol Table.—Continued.

SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL			SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL			SPECIFIC GRAVITY 20° C. / 4°	ALCOHOL		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.		Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.85436	**81.75**	*75.53*	64.53	0.84713	**84.25**	*78.50*	66.50	0.83957	**86.75**	*81.56*	68.48
0.85422	**81.80**	*75.59*	64.57	0.84698	**84.30**	*78.56*	66.54	0.83942	**86.80**	*81.62*	68.52
0.85408	**81.85**	*75.65*	64.61	0.84683	**84.35**	*78.62*	66.58	0.83927	**86.85**	*81.68*	68.56
0.85393	**81.90**	*75.71*	64.65	0.84668	**84.40**	*78.68*	66.62	0.83912	**86.90**	*81.75*	68.60
0.85379	**81.95**	*75.77*	64.69	0.84654	**84.45**	*78.74*	66.66	0.83896	**86.95**	*81.81*	68.64
0.85364	**82.00**	*75.82*	64.73	0.84639	**84.50**	*78.80*	66.70	0.83881	**87.00**	*81.87*	68.68
0.85350	**82.05**	*75.88*	64.77	0.84624	**84.55**	*78.86*	66.74	0.83865	**87.05**	*81.93*	68.72
0.85336	**82.10**	*75.94*	64.81	0.84609	**84.60**	*78.93*	66.78	0.83850	**87.10**	*81.99*	68.76
0.85322	**82.15**	*76.00*	64.85	0.84594	**84.65**	*78.99*	66.82	0.83834	**87.15**	*82.05*	68.80
0.85307	**82.20**	*76.06*	64.88	0.84579	**84.70**	*79.05*	66.86	0.83818	**87.20**	*81.12*	68.84
0.85293	**82.25**	*76.12*	64.92	0.84564	**84.75**	*79.11*	66.90	0.83802	**87.25**	*82.18*	68.88
0.85279	**82.30**	*76.18*	64.96	0.84549	**84.80**	*79.17*	66.94	0.83787	**87.30**	*82.24*	68.91
0.85265	**82.35**	*76.24*	65.00	0.84534	**84.85**	*79.23*	66.98	0.83771	**87.35**	*82.30*	68.95
0.85250	**82.40**	*76.30*	65.04	0.84519	**84.90**	*79.29*	67.02	0.83756	**87.40**	*82.37*	68.99
0.85236	**82.45**	*76.36*	65.08	0.84504	**84.95**	*79.35*	67.06	0.83740	**87.45**	*82.43*	69.03
0.85222	**82.50**	*76.41*	65.12	0.84489	**85.00**	*79.41*	67.09	0.83725	**87.50**	*82.49*	69.07
0.85207	**82.55**	*76.47*	65.16	0.84474	**85.05**	*79.47*	67.13	0.83709	**87.55**	*82.55*	69.11
0.85192	**82.60**	*76.53*	65.20	0.84459	**85.10**	*79.53*	67.17	0.83694	**87.60**	*82.62*	69.15
0.85178	**82.65**	*76.59*	65.24	0.84444	**85.15**	*79.59*	67.21	0.83678	**87.65**	*82.68*	69.19
0.85164	**82.70**	*76.65*	65.28	0.84429	**85.20**	*79.65*	67.25	0.83663	**87.70**	*82.74*	69.23
0.85150	**82.75**	*76.71*	65.32	0.84414	**85.25**	*79.71*	67.29	0.83647	**87.75**	*82.80*	69.27
0.85135	**82.80**	*76.77*	65.36	0.84399	**85.30**	*79.78*	67.33	0.83632	**87.80**	*82.87*	69.30
0.85121	**82.85**	*76.83*	65.40	0.84384	**85.35**	*79.84*	67.37	0.83616	**87.85**	*82.93*	69.34
0.85106	**82.90**	*76.89*	65.44	0.84369	**85.40**	*79.90*	67.41	0.83601	**87.90**	*82.99*	69.38
0.85092	**82.95**	*76.95*	65.48	0.84354	**85.45**	*79.96*	67.45	0.83585	**87.95**	*83.05*	69.42
0.85077	**83.00**	*77.01*	65.51	0.84339	**85.50**	*80.02*	67.49	0.83569	**88.00**	*83.12*	69.46
0.85063	**83.05**	*77.07*	65.55	0.84323	**85.55**	*80.08*	67.53	0.83553	**88.05**	*83.18*	69.50
0.85049	**83.10**	*77.13*	65.59	0.84308	**85.60**	*80.14*	67.57	0.83537	**88.10**	*83.25*	69.54
0.85035	**83.15**	*77.19*	65.63	0.84293	**85.65**	*80.20*	67.61	0.83521	**88.15**	*83.31*	69.58
0.85020	**83.20**	*77.24*	65.67	0.84278	**85.70**	*80.27*	67.65	0.83505	**88.20**	*83.37*	69.62
0.85006	**83.25**	*77.30*	65.71	0.84263	**85.75**	*80.33*	67.69	0.83489	**88.25**	*83.43*	69.66
0.84991	**83.30**	*77.36*	65.75	0.84248	**85.80**	*80.39*	67.73	0.83473	**88.30**	*83.50*	69.70
0.84977	**83.35**	*77.42*	65.79	0.84233	**85.85**	*80.45*	67.77	0.83457	**88.35**	*83.56*	69.74
0.84962	**83.40**	*77.48*	65.83	0.84218	**85.90**	*80.51*	67.80	0.83442	**88.40**	*83.63*	69.78
0.84948	**83.45**	*77.54*	65.87	0.84203	**85.95**	*80.57*	67.84	0.83426	**88.45**	*83.69*	69.82
0.84933	**83.50**	*77.60*	65.91	0.84188	**86.00**	*80.63*	67.88	0.83410	**88.50**	*83.75*	69.86
0.84918	**83.55**	*77.66*	65.95	0.84172	**86.05**	*80.69*	67.92	0.83394	**88.55**	*83.81*	69.90
0.84903	**83.60**	*77.72*	65.99	0.84157	**86.10**	*80.76*	67.96	0.83379	**88.60**	*83.88*	69.94
0.84889	**83.65**	*77.78*	66.03	0.84141	**86.15**	*80.82*	68.00	0.83363	**88.65**	*83.94*	69.98
0.84874	**83.70**	*77.84*	66.07	0.84126	**86.20**	*80.88*	68.04	0.83347	**88.70**	*84.00*	70.01
0.84859	**83.75**	*77.90*	66.11	0.84110	**86.25**	*80.94*	68.08	0.83331	**88.75**	*84.06*	70.05
0.84844	**83.80**	*77.96*	66.15	0.84095	**86.30**	*81.00*	68.12	0.83315	**88.80**	*84.13*	70.09
0.84830	**83.85**	*78.02*	66.19	0.84080	**86.35**	*81.06*	68.16	0.83299	**88.85**	*84.19*	70.13
0.84815	**83.90**	*78.08*	66.23	0.84065	**86.40**	*81.13*	68.20	0.83283	**88.90**	*84.26*	70.17
0.84801	**83.95**	*78.14*	66.27	0.84049	**86.45**	*81.19*	68.24	0.83267	**88.95**	*84.32*	70.21
0.84786	**84.00**	*78.20*	66.30	0.84034	**86.50**	*81.25*	68.28	0.83251	**89.00**	*84.39*	70.25
0.84772	**84.05**	*78.26*	66.34	0.84018	**86.55**	*81.31*	68.32	0.83235	**89.05**	*84.45*	70.29
0.84757	**84.10**	*78.32*	66.38	0.84003	**86.60**	*81.37*	68.36	0.83219	**89.10**	*84.51*	70.33
0.84742	**84.15**	*78.38*	66.42	0.83987	**86.65**	*81.43*	68.40	0.83203	**89.15**	*84.57*	70.37
0.84727	**84.20**	*78.44*	66.46	0.83972	**86.70**	*81.50*	68.44	0.83186	**89.20**	*84.64*	70.41

7

Alcohol Table.—Continued.

Specific gravity 20° C./4°	Alcohol		
	Per cent by volume at 20° C.	Per cent by weight	Grams per 100 cc.
0.83170	**89.25**	*84.70*	70.45
0.83154	**89.30**	*84.77*	70.49
0.83138	**89.35**	*84.83*	70.53
0.83121	**89.40**	*84.90*	70.57
0.83105	**89.45**	*84.96*	70.61
0.83089	**89.50**	*85.03*	70.65
0.83073	**89.55**	*85.09*	70.69
0.83056	**89.60**	*85.15*	70.72
0.83040	**89.65**	*85.21*	70.76
0.83024	**89.70**	*85.28*	70.80
0.83008	**89.75**	*85.34*	70.84
0.82991	**89.80**	*85.41*	70.88
0.82975	**89.85**	*85.47*	70.92
0.82958	**89.90**	*85.54*	70.96
0.82942	**89.95**	*85.60*	71.00
0.82925	**90.00**	*85.67*	71.04
0.82909	**90.05**	*85.73*	71.08
0.82892	**90.10**	*85.80*	71.12
0.82876	**90.15**	*85.86*	71.16
0.82859	**90.20**	*85.93*	71.20
0.82843	**90.25**	*85.99*	71.24
0.82826	**90.30**	*86.06*	71.28
0.82810	**90.35**	*86.12*	71.32
0.82793	**90.40**	*86.19*	71.36
0.82776	**90.45**	*86.25*	71.40
0.82759	**90.50**	*86.32*	71.44
0.82742	**90.55**	*86.38*	71.48
0.82725	**90.60**	*86.45*	71.52
0.82708	**90.65**	*86.51*	71.56
0.82691	**90.70**	*86.58*	71.59
0.82674	**90.75**	*86.64*	71.63
0.82657	**90.80**	*86.71*	71.67
0.82640	**90.85**	*86.77*	71.71
0.82624	**90.90**	*86.84*	71.75
0.82607	**90.95**	*86.90*	71.79
0.82590	**91.00**	*86.97*	71.83
0.82573	**91.05**	*87.03*	71.87
0.82556	**91.10**	*87.10*	71.91
0.82539	**91.15**	*87.17*	71.95
0.82522	**91.20**	*87.24*	71.99
0.82505	**91.25**	*87.30*	72.03
0.82488	**91.30**	*87.37*	72.07
0.82470	**91.35**	*87.43*	72.11
0.82453	**91.40**	*87.50*	72.15
0.82436	**91.45**	*87.56*	72.19
0.82419	**91.50**	*87.63*	72.23
0.82401	**91.55**	*87.69*	72.27
0.82384	**91.60**	*87.76*	72.30
0.82367	**91.65**	*87.83*	72.34
0.82350	**91.70**	*87.90*	72.38
0.82332	**91.75**	*87.96*	72.42
0.82315	**91.80**	*88.03*	72.46
0.82298	**91.85**	*88.09*	72.50
0.82281	**91.90**	*88.16*	72.54
0.82263	**91.95**	*88.22*	72.58
0.82246	**92.00**	*88.29*	72.62
0.82229	**92.05**	*88.36*	72.66
0.82212	**92.10**	*88.43*	72.70
0.82194	**92.15**	*88.49*	72.74
0.82177	**92.20**	*88.56*	72.78
0.82159	**92.25**	*88.63*	72.82
0.82141	**92.30**	*88.70*	72.86
0.82123	**92.35**	*88.76*	72.90
0.82106	**92.40**	*88.83*	72.94
0.82088	**92.45**	*88.89*	72.98
0.82071	**92.50**	*88.96*	73.02
0.82053	**92.55**	*89.03*	73.06
0.82035	**92.60**	*89.10*	73.09
0.82017	**92.65**	*89.16*	73.13
0.82000	**92.70**	*89.23*	73.17
0.81982	**92.75**	*89.30*	73.21
0.81964	**92.80**	*89.37*	73.25
0.81946	**92.85**	*89.43*	73.29
0.81929	**92.90**	*89.50*	73.33
0.81911	**92.95**	*89.57*	73.37
0.81893	**93.00**	*89.64*	73.41
0.81875	**93.05**	*89.71*	73.45
0.81856	**93.10**	*89.78*	73.49
0.81838	**93.15**	*89.84*	73.53
0.81821	**93.20**	*89.91*	73.57
0.81803	**93.25**	*89.98*	73.61
0.81784	**93.30**	*90.05*	73.65
0.81766	**93.35**	*90.12*	73.69
0.81748	**93.40**	*90.19*	73.72
0.81730	**93.45**	*90.25*	73.76
0.81711	**93.50**	*90.32*	73.80
0.81693	**93.55**	*90.39*	73.84
0.81675	**93.60**	*90.46*	73.88
0.81657	**93.65**	*90.53*	73.92
0.81638	**93.70**	*90.60*	73.96
0.81620	**93.75**	*90.67*	74.00
0.81601	**93.80**	*90.74*	74.04
0.81582	**93.85**	*90.80*	74.08
0.81563	**93.90**	*90.87*	74.12
0.81545	**93.95**	*90.94*	74.16
0.81526	**94.00**	*91.01*	74.20
0.81507	**94.05**	*91.08*	74.24
0.81488	**94.10**	*91.15*	74.28
0.81469	**94.15**	*91.22*	74.32
0.81450	**94.20**	*91.29*	74.36
0.81432	**94.25**	*91.36*	74.40
0.81413	**94.30**	*91.43*	74.44
0.81394	**94.35**	*91.50*	74.48
0.81375	**94.40**	*91.57*	74.52
0.81356	**94.45**	*91.64*	74.56
0.81337	**94.50**	*91.71*	74.59
0.81318	**94.55**	*91.78*	74.63
0.81299	**94.60**	*91.85*	74.67
0.81280	**94.65**	*91.92*	74.71
0.81260	**94.70**	*91.99*	74.75
0.81241	**94.75**	*92.06*	74.79
0.81222	**94.80**	*92.13*	74.83
0.81202	**94.85**	*92.20*	74.87
0.81183	**94.90**	*92.27*	74.91
0.81163	**94.95**	*92.34*	74.95
0.81144	**95.00**	*92.41*	74.99
0.81124	**95.05**	*92.48*	75.03
0.81105	**95.10**	*92.55*	75.07
0.81086	**95.15**	*92.63*	75.11
0.81067	**95.20**	*92.70*	75.15
0.81047	**95.25**	*92.77*	75.19
0.81028	**95.30**	*92.84*	75.23
0.81008	**95.35**	*92.91*	75.27
0.80988	**95.40**	*92.98*	75.30
0.80968	**95.45**	*93.05*	75.34
0.80949	**95.50**	*93.12*	75.38
0.80929	**95.55**	*93.20*	75.42
0.80909	**95.60**	*93.27*	75.46
0.80889	**95.65**	*93.34*	75.50
0.80869	**95.70**	*93.41*	75.54
0.80849	**95.75**	*93.48*	75.58
0.80829	**95.80**	*93.55*	75.62
0.80809	**95.85**	*93.63*	75.66
0.80789	**95.90**	*93.70*	75.70
0.80769	**95.95**	*93.77*	75.74
0.80749	**96.00**	*93.84*	75.78
0.80729	**96.05**	*93.92*	75.82
0.80709	**96.10**	*93.99*	75.86
0.80689	**96.15**	*94.06*	75.90
0.80668	**96.20**	*94.13*	75.94
0.80648	**96.25**	*94.21*	75.98
0.80627	**96.30**	*94.28*	76.01
0.80607	**96.35**	*94.35*	76.05
0.80586	**96.40**	*94.42*	76.09
0.80566	**96.45**	*94.50*	76.13
0.80545	**96.50**	*94.57*	76.17
0.80525	**96.55**	*94.65*	76.21
0.80504	**96.60**	*94.72*	76.25
0.80483	**96.65**	*94.79*	76.29
0.80462	**96.70**	*94.86*	76.33

7

Alcohol Table.—Concluded.

SPECIFIC GRAVITY $\frac{20°C.}{4°}$	ALCOHOL		
	Per cent by volume at 20°C.	Per cent by weight	Grams per 100 cc.
0.80442	96.75	94.94	76.37
0.80421	96.80	95.01	76.41
0.80400	96.85	95.09	76.45
0.80379	96.90	95.16	76.49
0.80358	96.95	95.24	76.53
0.80337	97.00	95.31	76.57
0.80315	97.05	95.39	76.61
0.80294	97.10	95.46	76.65
0.80273	97.15	95.53	76.69
0.80252	97.20	95.60	76.72
0.80230	97.25	95.68	76.76
0.80208	97.30	95.75	76.80
0.80186	97.35	95.83	76.84
0.80164	97.40	95.91	76.88
0.80143	97.45	95.98	76.92
0.80122	97.50	96.05	76.96
0.80100	97.55	96.13	77.00
0.80078	97.60	96.21	77.04
0.80056	97.65	96.29	77.08
0.80034	97.70	96.36	77.12
0.80012	97.75	96.44	77.16
0.79990	97.80	96.52	77.20
0.79968	97.85	96.60	77.24
0.79945	97.90	96.68	77.28
0.79923	97.95	96.75	77.32
0.79900	98.00	96.82	77.36
0.79878	98.05	96.90	77.40
0.79855	98.10	96.97	77.43
0.79832	98.15	97.05	77.47
0.79809	98.20	97.12	77.51
0.79786	98.25	97.20	77.55
0.79763	98.30	97.28	77.59
0.79740	98.35	97.36	77.63
0.79717	98.40	97.43	77.67
0.79695	98.45	97.51	77.71
0.79672	98.50	97.59	77.75
0.79648	98.55	97.67	77.79
0.79625	98.60	97.75	77.83
0.79601	98.65	97.83	77.87
0.79577	98.70	97.90	77.91
0.79553	98.75	97.98	77.95
0.79529	98.80	98.06	77.99
0.79505	98.85	98.14	78.03
0.79481	98.90	98.22	78.07
0.79457	98.95	98.30	78.11
0.79432	99.00	98.38	78.14
0.79408	99.05	98.46	78.18
0.79384	99.10	98.54	78.22
0.79360	99.15	98.62	78.26
0.79335	99.20	98.70	78.30
0.79311	99.25	98.78	78.34
0.79286	99.30	98.86	78.38
0.79262	99.35	98.94	78.42
0.79237	99.40	99.02	78.46
0.79213	99.45	99.10	78.50
0.79188	99.50	99.18	78.54
0.79163	99.55	99.26	78.58
0.79138	99.60	99.34	78.62
0.79113	99.65	99.42	78.66
0.79088	99.70	99.51	78.70
0.79062	99.75	99.59	78.74
0.79037	99.80	99.67	78.78
0.79011	99.85	99.75	78.82
0.78986	99.90	99.83	78.86
0.78960	99.95	99.91	78.90
0.78934	100.00	100.00	78.93

8 **ALCOHOL**

For calculating the percentages of alcohol in mixtures of ethyl alcohol and

SCALE READING	17.5°C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
13.2	----	----	----	----	----	----	----	----	----
13.3	----	----	----	----	----	----	----	----	----
13.4	----	----	----	----	----	----	----	----	----
13.5	----	----	----	----	----	----	----	----	----
13.6	----	----	----	----	----	----	----	----	----
13.7	----	----	----	----	----	----	----	----	----
13.8	----	----	----	----	----	----	----	----	----
13.9	----	----	----	----	----	----	----	----	----
14.0	----	----	----	----	----	----	----	----	----
14.1	----	----	----	----	----	----	----	----	----
14.2	----	----	----	----	----	----	----	----	0.04
14.3	----	----	----	----	----	----	----	----	0.13
14.4	----	----	----	----	----	----	----	----	0.21
14.5	----	----	----	----	----	----	0.08	*0.06*	0.29
14.6	----	----	----	----	----	----	0.16	*0.13*	0.38
14.7	----	----	----	----	0.05	*0.04*	0.25	*0.20*	0.46
14.8	----	----	----	----	0.14	*0.11*	0.34	*0.27*	0.55
14.9	----	----	0.01	*0.01*	0.23	*0.18*	0.43	*0.34*	0.64
15.0	0.00	*0.00*	0.10	*0.08*	0.31	*0.24*	0.52	*0.41*	0.73
15.1	0.09	*0.07*	0.19	*0.15*	0.39	*0.31*	0.60	*0.48*	0.82
15.2	0.17	*0.13*	0.27	*0.21*	0.48	*0.38*	0.69	*0.55*	0.91
15.3	0.25	*0.20*	0.35	*0.28*	0.57	*0.45*	0.77	*0.61*	0.99
15.4	0.34	*0.27*	0.44	*0.35*	0.65	*0.51*	0.85	*0.68*	1.07
15.5	0.43	*0.34*	0.53	*0.42*	0.73	*0.58*	0.94	*0.75*	1.16
15.6	0.51	*0.40*	0.60	*0.48*	0.82	*0.65*	1.03	*0.82*	1.24
15.7	0.59	*0.47*	0.69	*0.55*	0.91	*0.72*	1.12	*0.89*	1.32
15.8	0.68	*0.54*	0.78	*0.62*	0.99	*0.79*	1.21	*0.96*	1.40
15.9	0.76	*0.60*	0.85	*0.68*	1.08	*0.86*	1.28	*1.02*	1.47
16.0	0.84	*0.67*	0.94	*0.75*	1.17	*0.93*	1.36	*1,08*	1.55
16.1	0.93	*0.74*	1.03	*0.82*	1.24	*0.99*	1.44	*1.14*	1.62
16.2	1.02	*0.81*	1.12	*0.89*	1.32	*1.05*	1.51	*1.20*	1.70
16.3	1.10	*0.87*	1.19	*0.95*	1.40	*1.11*	1.59	*1.26*	1.77
16.4	1.18	*0.94*	1.29	*1.02*	1.47	*1.17*	1.66	*1.32*	1.85
16.5	1.26	*1.00*	1.36	*1.08*	1.55	*1.23*	1.74	*1.38*	1.92
16.6	1.34	*1.06*	1.43	*1.13*	1.62	*1.29*	1.81	*1.44*	2.00
16.7	1.41	*1.12*	1.50	*1.19*	1.70	*1.35*	1.89	*1.50*	2.07
16.8	1.49	*1.18*	1.57	*1.25*	1.77	*1.41*	1.96	*1.56*	2.15
16.9	1.56	*1.24*	1.65	*1.31*	1.85	*1.47*	2.04	*1.62*	2.22
17.0	1.63	*1.30*	1.72	*1.37*	1.92	*1.53*	2.11	*1.68*	2.30
17.1	1.70	*1.35*	1.80	*1.43*	1.99	*1.58*	2.19	*1.74*	2.37
17.2	1.77	*1.41*	1.87	*1.49*	2.06	*1,64*	2.26	*1.80*	2.45
17.3	1.85	*1.47*	1.94	*1.54*	2.14	*1.70*	2.34	*1.86*	2.52
17.4	1.92	*1.53*	2.01	*1.60*	2.21	*1.76*	2.41	*1.92*	2.59
17.5	2.00	*1.59*	2.09	*1.66*	2.29	*1.82*	2.49	*1.98*	2.66
17.6	2.07	*1.65*	2.16	*1.72*	2.36	*1.88*	2.56	*2.04*	2.74
17.7	2.14	*1.70*	2.24	*1.78*	2.44	*1.94*	2.62	*2.09*	2.81
17.8	2.21	*1.76*	2.31	*1.84*	2.51	*2.00*	2.70	*2.15*	2.89
17.9	2.29	*1.82*	2.38	*1.89*	2.59	*2.06*	2.77	*2.21*	2.96

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[a] Calculated and arranged by B. H. St. John from data of Doroshevskii and Dvorzhanchik.

TABLE. 8

water from their Zeiss immersion refractometer readings at 17.5°–25°C.

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
----	----	----	----	----	----	----	0.00	0.00	13.2
----	----	----	----	----	----	----	0.09	0.07	13.3
----	----	----	----	----	----	----	0.18	0.14	13.4
----	----	----	----	----	0.05	0.04	0.26	0.21	13.5
----	----	----	----	----	0.14	0.11	0.35	0.28	13.6
----	----	----	0.01	0.01	0.23	0.18	0.44	0.35	13.7
----	----	----	0.10	0.08	0.31	0.25	0.53	0.42	13.8
----	----	----	0.19	0.15	0.40	0.32	0.62	0.49	13.9
----	0.08	0.06	0.28	0.22	0.49	0.39	0.70	0.56	14.0
----	0.16	0.13	0.36	0.29	0.58	0.46	0.79	0.63	14.1
0.03	0.24	0.19	0.45	0.36	0.67	0.53	0.88	0.70	14.2
0.10	0.33	0.26	0.54	0.43	0.75	0.60	0.97	0.77	14.3
0.17	0.41	0.33	0.63	0.50	0.84	0.67	1.06	0.85	14.4
0.23	0.50	0.40	0.72	0.57	0.93	0.74	1.15	0.92	14.5
0.30	0.59	0.47	0.80	0.64	1.02	0.81	1.24	0.99	14.6
0.37	0.68	0.54	0.89	0.71	1.11	0.88	1.32	1.05	14.7
0.44	0.77	0.61	0.98	0.78	1.19	0.95	1.40	1.11	14.8
0.51	0.85	0.68	1.07	0.85	1.28	1.02	1.47	1.17	14.9
0.58	0.94	0.75	1.16	0.92	1.36	1.08	1.55	1.23	15.0
0.65	1.03	0.82	1.24	0.99	1.44	1.14	1.63	1.29	15.1
0.72	1.12	0.89	1.32	1.05	1.51	1.20	1.71	1.36	15.2
0.79	1.21	0.96	1.40	1.11	1.59	1.26	1.79	1.42	15.3
0.85	1.29	1.02	1.47	1.17	1.66	1.32	1.86	1.48	15.4
0.92	1.36	1.08	1.55	1.23	1.74	1.38	1.94	1.54	15.5
0.99	1.44	1.15	1.62	1.29	1.82	1.44	2.01	1.60	15.6
1.05	1.52	1.21	1.70	1.35	1.90	1.51	2.09	1.66	15.7
1.11	1.60	1.27	1.77	1.41	1.97	1.57	2.17	1.72	15.8
1.17	1.67	1.33	1.85	1.47	2.05	1.63	2.25	1.79	15.9
1.23	1.75	1.39	1.92	1.53	2.12	1.69	2.33	1.85	16.0
1.29	1.82	1.45	2.00	1.59	2.20	1.75	2.40	1.91	16.1
1.35	1.90	1.51	2.08	1.65	2.27	1.81	2.48	1.97	16.2
1.41	1.97	1.57	2.16	1.72	2.35	1.87	2.55	2.03	16.3
1.47	2.05	1.63	2.24	1.78	2.43	1.93	2.62	2.09	16.4
1.53	2.12	1.69	2.31	1.84	2.50	1.99	2.70	2.15	16.5
1.59	2.20	1.75	2.39	1.90	2.57	2.05	2.77	2.21	16.6
1.65	2.27	1.81	2.46	1.96	2.65	2.11	2.85	2.27	16.7
1.71	2.35	1.87	2.53	2.02	2.72	2.17	2.92	2.33	16.8
1.77	2.43	1.93	2.61	2.08	2.80	2.23	2.99	2.38	16.9
1.83	2.50	1.99	2.69	2.14	2.87	2.29	3.06	2.44	17.0
1.89	2.57	2.05	2.76	2.20	2.95	2.35	3.14	2.50	17.1
1.95	2.65	2.11	2.82	2.25	3.02	2.41	3.21	2.56	17.2
2.01	2.72	2.17	2.90	2.31	3.10	2.47	3.29	2.62	17.3
2.07	2.79	2.23	2.97	2.37	3.17	2.53	3.36	2.68	17.4
2.12	2.86	2.28	3.04	2.43	3.25	2.59	3.43	2.74	17.5
2.18	2.94	2.34	3.12	2.49	3.32	2.65	3.51	2.80	17.6
2.24	3.01	2.40	3.20	2.55	3.39	2.70	3.58	2.86	17.7
2.30	3.09	2.46	3.27	2.61	3.46	2.76	3.66	2.92	17.8
2.36	3.16	2.52	3.35	2.67	3.53	2.82	3.73	2.98	17.9

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
18.0	2.36	*1.88*	2.45	*1.95*	2.66	*2.12*	2.85	*2.27*	3.04
18.1	2.43	*1.94*	2.52	*2.01*	2.74	*2.18*	2.92	*2.33*	3.11
18.2	2.50	*2.00*	2.60	*2.07*	2.81	*2.24*	3.00	*2.39*	3.19
18.3	2.57	*2.05*	2.67	*2.13*	2.89	*2.30*	3.07	*2.45*	3.26
18.4	2.65	*2.11*	2.75	*2.19*	2.96	*2.36*	3.15	*2.51*	3.34
18.5	2.72	*2.17*	2.82	*2.25*	3.03	*2.41*	3.22	*2.57*	3.41
18.6	2.80	*2.23*	2.90	*2.31*	3.10	*2.47*	3.30	*2.63*	3.48
18.7	2.87	*2.29*	2.97	*2.37*	3.17	*2.53*	3.37	*2.69*	3.55
18.8	2.95	*2.35*	3.05	*2.43*	3.25	*2.59*	3.45	*2.75*	3.63
18.9	3.02	*2.41*	3.12	*2.49*	3.32	*2.65*	3.52	*2.81*	3.70
19.0	3.10	*2.47*	3.19	*2.54*	3.40	*2.71*	3.59	*2.86*	3.77
19.1	3.17	*2.53*	3.26	*2.60*	3.47	*2.77*	3.66	*2.92*	3.85
19.2	3.25	*2.59*	3.34	*2.66*	3.55	*2.83*	3.73	*2.98*	3.92
19.3	3.32	*2.65*	3.41	*2.72*	3.62	*2.89*	3.81	*3.04*	4.00
19.4	3.39	*2.70*	3.48	*2.78*	3.70	*2.95*	3.88	*3.10*	4.07
19.5	3.46	*2.76*	3.56	*2.84*	3.77	*3.01*	3.96	*3.16*	4.14
19.6	3.53	*2.82*	3.63	*2.90*	3.84	*3.06*	4.03	*3.22*	4.22
19.7	3.61	*2.88*	3.71	*2.96*	3.91	*3.12*	4.10	*3.27*	4.29
19.8	3.68	*2.94*	3.78	*3.02*	3.98	*3.18*	4.17	*3.33*	4.37
19.9	3.76	*3.00*	3.86	*3.08*	4.06	*3.24*	4.25	*3.39*	4.44
20.0	3.83	*3.06*	3.93	*3.13*	4.13	*3.30*	4.32	*3.45*	4.52
20.1	3.90	*3.12*	4.00	*3.19*	4.20	*3.35*	4.39	*3.51*	4.59
20.2	3.97	*3.17*	4.07	*3.25*	4.27	*3.41*	4.47	*3.57*	4.66
20.3	4.04	*3.23*	4.14	*3.31*	4.34	*3.47*	4.54	*3.63*	4.74
20.4	4.12	*3.29*	4.22	*3.37*	4.42	*3.53*	4.61	*3.68*	4.82
20.5	4.19	*3.35*	4.29	*3.43*	4.49	*3.59*	4.68	*3.74*	4.89
20.6	4.26	*3.41*	4.36	*3.49*	4.56	*3.65*	4.75	*3.80*	4.96
20.7	4.33	*3.46*	4.43	*3.54*	4.63	*3.70*	4.83	*3.86*	5.03
20.8	4.41	*3.52*	4.51	*3.60*	4.70	*3.76*	4.90	*3.92*	5.10
20.9	4.48	*3.58*	4.58	*3.66*	4.78	*3.82*	4.97	*3.98*	5.17
21.0	4.56	*3.64*	4.65	*3.72*	4.85	*3.88*	5.04	*4.03*	5.24
21.1	4.63	*3.70*	4.73	*3.78*	4.92	*3.94*	5.11	*4.09*	5.31
21.2	4.70	*3.76*	4.80	*3.84*	4.99	*3.99*	5.19	*4.15*	5.39
21.3	4.77	*3.81*	4.87	*3.89*	5.06	*4.05*	5.26	*4.21*	5.46
21.4	4.84	*3.87*	4.94	*3.95*	5.14	*4.11*	5.33	*4.26*	5.53
21.5	4.92	*3.93*	5.01	*4.01*	5.21	*4.17*	5.40	*4.32*	5.60
21.6	4.99	*3.99*	5.09	*4.07*	5.28	*4.22*	5.47	*4.38*	5.67
21.7	5.06	*4.05*	5.16	*4.13*	5.35	*4.28*	5.54	*4.44*	5.75
21.8	5.13	*4.10*	5.23	*4.18*	5.43	*4.34*	5.61	*4.49*	5.82
21.9	5.20	*4.16*	5.30	*4.24*	5.50	*4.40*	5.69	*4.55*	5.89
22.0	5.27	*4.22*	5.37	*4.30*	5.57	*4.45*	5.76	*4.61*	5.96
22.1	5.34	*4.27*	5.44	*4.35*	5.64	*4.51*	5.83	*4.67*	6.03
22.2	5.41	*4.33*	5.51	*4.41*	5.71	*4.57*	5.90	*4.72*	6.11
22.3	5.49	*4.39*	5.58	*4.47*	5.78	*4.63*	5.97	*4.78*	6.18
22.4	5.56	*4.45*	5.65	*4.53*	5.85	*4.68*	6.05	*4.84*	6.25
22.5	5.63	*4.51*	5.72	*4.58*	5.92	*4.74*	6.12	*4.90*	6.32
22.6	5.70	*4.56*	5.80	*4.64*	6.00	*4.80*	6.19	*4.95*	6.39
22.7	5.77	*4.62*	5.87	*4.70*	6.07	*4.86*	6.26	*5.01*	6.46
22.8	5.85	*4.68*	5.94	*4.75*	6.14	*4.91*	6.33	*5.07*	6.53
22.9	5.92	*4.74*	6.01	*4.81*	6.21	*4.97*	6.40	*5.13*	6.60

TABLE.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
2.42	3.23	*2.58*	3.42	*2.73*	3.61	*2.88*	3.81	*3.04*	18.0
2.48	3.30	*2.63*	3.50	*2.79*	3.68	*2.94*	3.88	*3.10*	18.1
2.54	3.37	*2.69*	3.57	*2.85*	3.76	*3.00*	3.96	*3.16*	18.2
2.60	3.45	*2.75*	3.64	*2.91*	3.83	*3.06*	4.03	*3.22*	18.3
2.66	3.52	*2.81*	3.71	*2.96*	3.91	*3.12*	4.11	*3.28*	18.4
2.72	3.59	*2.87*	3.78	*3.02*	3.98	*3.18*	4.18	*3.34*	18.5
2.78	3.66	*2.92*	3.86	*3.08*	4.06	*3.24*	4.26	*3.40*	18.6
2.83	3.73	*2.98*	3.93	*3.14*	4.13	*3.30*	4.33	*3.46*	18.7
2.89	3.81	*3.04*	4.01	*3.20*	4.21	*3.36*	4.41	*3.52*	18.8
2.95	3.88	*3.10*	4.08	*3.26*	4.28	*3.42*	4.48	*3.58*	18.9
3.01	3.96	*3.16*	4.16	*3.32*	4.36	*3.48*	4.56	*3.64*	19.0
3.07	4.03	*3.22*	4.23	*3.38*	4.43	*3.54*	4.63	*3.70*	19.1
3.13	4.11	*3.28*	4.31	*3.44*	4.51	*3.60*	4.70	*3.76*	19.2
3.19	4.18	*3.34*	4.38	*3.50*	4.58	*3.66*	4.78	*3.82*	19.3
3.25	4.26	*3.40*	4.46	*3.56*	4.65	*3.72*	4.85	*3.88*	19.4
3.31	4.33	*3.46*	4.53	*3.62*	4.73	*3.78*	4.93	*3.94*	19.5
3.37	4.41	*3.52*	4.61	*3.68*	4.80	*3.84*	5.00	*4.00*	19.6
3.43	4.48	*3.58*	4.68	*3.74*	4.88	*3.90*	5.08	*4.06*	19.7
3.49	4.56	*3.64*	4.75	*3.80*	4.95	*3.96*	5.15	*4.12*	19.8
3.55	4.63	*3.70*	4.83	*3.86*	5.03	*4.02*	5.22	*4.17*	19.9
3.61	4.72	*3.77*	4.90	*3.92*	5.10	*4.08*	5.29	*4.23*	20.0
3.67	4.79	*3.83*	4.98	*3.98*	5.17	*4.13*	5.36	*4.29*	20.1
3.73	4.87	*3.89*	5.05	*4.04*	5.24	*4.19*	5.44	*4.35*	20.2
3.79	4.94	*3.95*	5.13	*4.10*	5.31	*4.25*	5.51	*4.41*	20.3
3.85	5.01	*4.01*	5.20	*4.16*	5.38	*4.31*	5.58	*4.47*	20.4
3.91	5.08	*4.06*	5.27	*4.21*	5.45	*4.37*	5.65	*4.52*	20.5
3.97	5.15	*4.12*	5.34	*4.27*	5.52	*4.42*	5.72	*4.58*	20.6
4.02	5.22	*4.18*	5.41	*4.33*	5.60	*4.48*	5.80	*4.64*	20.7
4.08	5.29	*4.24*	5.48	*4.39*	5.67	*4.54*	5.87	*4.70*	20.8
4.14	5.36	*4.29*	5.55	*4.45*	5.75	*4.60*	5.95	*4.76*	20.9
4.20	5.44	*4.35*	5.62	*4.50*	5.82	*4.66*	6.02	*4.81*	21.0
4.25	5.51	*4.41*	5.70	*4.56*	5.89	*4.72*	6.09	*4.87*	21.1
4.31	5.58	*4.47*	5.77	*4.62*	5.96	*4.77*	6.16	*4.93*	21.2
4.37	5.65	*4.52*	5.84	*4.68*	6.03	*4.83*	6.23	*4.99*	21.3
4.43	5.72	*4.58*	5.91	*4.73*	6.11	*4.89*	6.30	*5.05*	21.4
4.48	5.80	*4.64*	5.98	*4.79*	6.18	*4.95*	6.37	*5.10*	21.5
4.54	5.87	*4.70*	6.06	*4.85*	6.25	*5.01*	6.44	*5.16*	21.6
4.60	5.94	*4.75*	6.13	*4.91*	6.32	*5.06*	6.52	*5.22*	21.7
4.66	6.01	*4.81*	6.20	*4.97*	6.39	*5.12*	6.59	*5.28*	21.8
4.71	6.08	*4.87*	6.27	*5.02*	6.47	*5.18*	6.66	*5.34*	21.9
4.77	6.15	*4.93*	6.34	*5.08*	6.54	*5.24*	6.73	*5.39*	22.0
4.83	6.22	*4.98*	6.42	*5.14*	6.61	*5.29*	6.80	*5.45*	22.1
4.89	6.29	*5.04*	6.49	*5.20*	6.68	*5.35*	6.87	*5.51*	22.2
4.95	6.36	*5.10*	6.56	*5.25*	6.75	*5.41*	6.94	*5.57*	22.3
5.00	6.43	*5.15*	6.63	*5.31*	6.82	*5.47*	7.01	*5.62*	22.4
5.06	6.50	*5.21*	6.70	*5.37*	6.89	*5.52*	7.08	*5.68*	22.5
5.11	6.57	*5.27*	6.77	*5.43*	6.96	*5.58*	7.16	*5.74*	22.6
5.17	6.64	*5.33*	6.84	*5.48*	7.03	*5.64*	7.23	*5.80*	22.7
5.23	6.71	*5.38*	6.91	*5.54*	7.10	*5.70*	7.31	*5.86*	22.8
5.29	6.78	*5.44*	6.99	*5.60*	7.17	*5.75*	7.38	*5.91*	22.9

8 ALCOHOL

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
23.0	5.99	4.79	6.08	4.87	6.28	5.03	6.47	5.18	6.67
23.1	6.06	4.85	6.15	4.93	6.35	5.09	6.54	5.24	6.74
23.2	6.13	4.91	6.22	4.98	6.42	5.14	6.61	5.30	6.81
23.3	6.20	4.97	6.29	5.04	6.49	5.20	6.68	5.36	6.88
23.4	6.27	5.02	6.36	5.10	6.56	5.26	6.75	5.41	6.95
23.5	6.34	5.08	6.43	5.15	6.63	5.31	6.83	5.47	7.02
23.6	6.41	5.14	6.50	5.21	6.70	5.37	6.90	5.53	7.09
23.7	6.48	5.19	6.57	5.27	6.78	5.43	6.97	5.59	7.16
23.8	6.55	5.25	6.64	5.32	6.85	5.49	7.04	5.64	7.23
23.9	6.62	5.30	6.71	5.38	6.92	5.54	7.11	5.70	7.31
24.0	6.69	5.36	6.78	5.44	6.99	5.60	7.18	5.76	7.38
24.1	6.76	5.42	6.85	5.49	7.06	5.66	7.25	5.82	7.45
24.2	6.83	5.47	6.92	5.55	7.13	5.71	7.32	5.87	7.52
24.3	6.90	5.53	6.99	5.61	7.20	5.77	7.39	5.93	7.59
24.4	6.97	5.59	7.06	5.66	7.27	5.83	7.46	5.99	7.66
24.5	7.04	5.64	7.13	5.72	7.34	5.89	7.53	6.04	7.73
24.6	7.11	5.70	7.20	5.78	7.41	5.94	7.60	6.10	7.80
24.7	7.18	5.76	7.27	5.83	7.48	6.00	7.67	6.15	7.86
24.8	7.25	5.81	7.35	5.89	7.55	6.06	7.74	6.21	7.93
24.9	7.32	5.87	7.42	5.95	7.62	6.11	7.81	6.26	8.00
25.0	7.39	5.93	7.49	6.01	7.68	6.16	7.88	6.32	8.06
25.1	7.46	5.98	7.56	6.06	7.75	6.22	7.94	6.37	8.13
25.2	7.53	6.04	7.63	6.12	7.82	6.27	8.01	6.43	8.20
25.3	7.59	6.09	7.69	6.17	7.89	6.33	8.07	6.48	8.27
25.4	7.66	6.15	7.76	6.23	7.95	6.38	8.14	6.54	8.34
25.5	7.73	6.20	7.83	6.28	8.02	6.44	8.21	6.59	8.41
25.6	7.80	6.26	7.90	6.34	8.09	6.49	8.28	6.65	8.48
25.7	7.87	6.31	7.96	6.39	8.16	6.55	8.35	6.70	8.55
25.8	7.94	6.37	8.03	6.44	8.22	6.60	8.42	6.76	8.62
25.9	8.00	6.42	8.10	6.50	8.29	6.66	8.48	6.81	8.69
26.0	8.07	6.48	8.16	6.55	8.36	6.71	8.55	6.87	8.75
26.1	8.14	6.53	8.23	6.61	8.43	6.77	8.62	6.92	8.82
26.2	8.21	6.59	8.30	6.66	8.50	6.82	8.69	6.98	8.89
26.3	8.27	6.64	8.37	6.72	8.57	6.88	8.75	7.03	8.96
26.4	8.34	6.70	8.44	6.78	8.63	6.93	8.82	7.09	9.03
26.5	8.41	6.75	8.50	6.83	8.70	6.99	8.89	7.15	9.10
26.6	8.48	6.81	8.57	6.88	8.77	7.04	8.96	7.20	9.16
26.7	8.55	6.86	8.64	6.94	8.84	7.10	9.03	7.26	9.23
26.8	8.62	6.92	8.71	6.99	8.91	7.15	9.10	7.31	9.30
26.9	8.68	6.97	8.78	7.05	8.98	7.21	9.17	7.37	9.37
27.0	8.75	7.03	8.85	7.11	9.05	7.27	9.23	7.42	9.44
27.1	8.82	7.08	8.91	7.16	9.11	7.32	9.30	7.48	9.51
27.2	8.89	7.14	8.98	7.22	9.18	7.38	9.37	7.54	9.58
27.3	8.95	7.19	9.05	7.27	9.25	7.43	9.44	7.59	9.65
27.4	9.02	7.25	9.12	7.33	9.32	7.49	9.51	7.65	9.71
27.5	9.09	7.30	9.19	7.38	9.38	7.54	9.58	7.70	9.78
27.6	9.16	7.36	9.26	7.44	9.45	7.60	9.65	7.76	9.85
27.7	9.22	7.41	9.32	7.49	9.52	7.65	9.72	7.82	9.91
27.8	9.29	7.47	9.39	7.55	9.59	7.71	9.79	7.87	9.98
27.9	9.36	7.52	9.46	7.60	9.65	7.76	9.86	7.93	10.05

TABLE.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		SCALE READING
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	
5.34	6.86	*5.50*	7.06	*5.66*	7.24	*5.81*	7.45	*5.97*	23.0
5.40	6.93	*5.56*	7.13	*5.71*	7.32	*5.87*	7.52	*6.03*	23.1
5.46	7.00	*5.61*	7.20	*5.77*	7.39	*5.93*	7.59	*6.08*	23.2
5.51	7.07	*5.67*	7.27	*5.83*	7.46	*5.98*	7.66	*6.14*	23.3
5.57	7.14	*5.73*	7.34	*5.89*	7.53	*6.04*	7.73	*6.20*	23.4
5.63	7.21	*5.78*	7.41	*5.94*	7.60	*6.10*	7.80	*6.25*	23.5
5.69	7.28	*5.84*	7.48	*6.00*	7.67	*6.15*	7.87	*6.31*	23.6
5.74	7.35	*5.89*	7.55	*6.06*	7.74	*6.21*	7.94	*6.37*	23.7
5.80	7.42	*5.95*	7.62	*6.11*	7.81	*6.27*	8.00	*6.42*	23.8
5.86	7.49	*6.01*	7.69	*6.17*	7.88	*6.32*	8.07	*6.48*	23.9
5.92	7.56	*6.07*	7.76	*6.22*	7.95	*6.38*	8.14	*6.53*	24.0
5.97	7.63	*6.12*	7.83	*6.28*	8.02	*6.44*	8.21	*6.59*	24.1
6.03	7.70	*6.18*	7.90	*6.34*	8.09	*6.49*	8.28	*6.65*	24.2
6.08	7.77	*6.24*	7.97	*6.39*	8.16	*6.55*	8.35	*6.70*	24.3
6.14	7.84	*6.29*	8.04	*6.45*	8.23	*6.60*	8.42	*6.76*	24.4
6.20	7.91	*6.35*	8.10	*6.50*	8.30	*6.66*	8.48	*6.81*	24.5
6.25	7.98	*6.41*	8.17	*6.56*	8.37	*6.72*	8.55	*6.87*	24.6
6.31	8.05	*6.46*	8.24	*6.62*	8.44	*6.77*	8.62	*6.93*	24.7
6.36	8.12	*6.52*	8.31	*6.67*	8.51	*6.83*	8.69	*6.98*	24.8
6.42	8.19	*6.58*	8.38	*6.73*	8.58	*6.89*	8.76	*7.04*	24.9
6.47	8.26	*6.63*	8.45	*6.79*	8.64	*6.94*	8.84	*7.10*	25.0
6.53	8.33	*6.69*	8.52	*6.84*	8.71	*7.00*	8.91	*7.15*	25.1
6.59	8.40	*6.75*	8.59	*6.90*	8.78	*7.06*	8.98	*7.21*	25.2
6.64	8.47	*6.80*	8.66	*6.96*	8.85	*7.11*	9.05	*7.27*	25.3
6.70	8.54	*6.86*	8.73	*7.01*	8.92	*7.17*	9.12	*7.33*	25.4
6.75	8.61	*6.92*	8.80	*7.07*	8.99	*7.23*	9.19	*7.38*	25.5
6.81	8.68	*6.97*	8.86	*7.12*	9.06	*7.28*	9.26	*7.44*	25.6
6.87	8.75	*7.03*	8.93	*7.18*	9.13	*7.34*	9.33	*7.50*	25.7
6.92	8.82	*7.08*	9.00	*7.23*	9.20	*7.40*	9.39	*7.55*	25.8
6.98	8.89	*7.14*	9.07	*7.29*	9.27	*7.45*	9.46	*7.61*	25.9
7.03	8.95	*7.19*	9.14	*7.35*	9.34	*7.51*	9.53	*7.67*	26.0
7.09	9.02	*7.25*	9.21	*7.40*	9.41	*7.56*	9.60	*7.73*	26.1
7.14	9.09	*7.30*	9.28	*7.46*	9.48	*7.62*	9.67	*7.78*	26.2
7.20	9.16	*7.36*	9.35	*7.51*	9.55	*7.68*	9.74	*7.84*	26.3
7.25	9.22	*7.41*	9.42	*7.57*	9.61	*7.73*	9.81	*7.90*	26.4
7.31	9.29	*7.47*	9.49	*7.63*	9.68	*7.79*	9.88	*7.95*	26.5
7.36	9.36	*7.52*	9.55	*7.68*	9.75	*7.85*	9.95	*8.01*	26.6
7.42	9.43	*7.58*	9.62	*7.74*	9.82	*7.90*	10.02	*8.07*	26.7
7.47	9.49	*7.63*	9.69	*7.79*	9.89	*7.96*	10.09	*8.12*	26.8
7.53	9.56	*7.69*	9.76	*7.85*	9.96	*8.02*	10.16	*8.18*	26.9
7.59	9.63	*7.74*	9.83	*7.91*	10.03	*8.07*	10.23	*8.24*	27.0
7.65	9.70	*7.80*	9.90	*7.96*	10.10	*8.13*	10.30	*8.29*	27.1
7.70	9.76	*7.85*	9.97	*8.02*	10.17	*8.18*	10.37	*8.35*	27.2
7.76	9.83	*7.91*	10.03	*8.07*	10.24	*8.24*	10.44	*8.40*	27.3
7.81	9.90	*7.96*	10.10	*8.13*	10.31	*8.30*	10.51	*8.46*	27.4
7.86	9.97	*8.02*	10.17	*8.18*	10.38	*8.35*	10.58	*8.52*	27.5
7.92	10.03	*8.07*	10.24	*8.24*	10.45	*8.41*	10.65	*8.57*	27.6
7.97	10.10	*8.13*	10.31	*8.30*	10.51	*8.46*	10.72	*8.63*	27.7
8.03	10.17	*8.18*	10.38	*8.35*	10.58	*8.52*	10.79	*8.69*	27.8
8.08	10.24	*8.24*	10.45	*8.41*	10.65	*8.58*	10.86	*8.74*	27.9

8 ALCOHOL

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
28.0	9.43	7.58	9.53	7.66	9.72	7.82	9.92	7.98	10.12
28.1	9.50	7.64	9.59	7.71	9.79	7.87	9.99	8.04	10.18
28.2	9.57	7.69	9.66	7.77	9.86	7.93	10.06	8.09	10.25
28.3	9.64	7.75	9.73	7.82	9.92	7.98	10.13	8.15	10.32
28.4	9.70	7.80	9.80	7.88	9.99	8.04	10.19	8.20	10.39
28.5	9.77	7.86	9.86	7.93	10.06	8.09	10.26	8.26	10.45
28.6	9.84	7.91	9.93	7.99	10.13	8.15	10.32	8.31	10.52
28.7	9.91	7.97	10.00	8.04	10.19	8.20	10.39	8.36	10.59
28.8	9.97	8.02	10.07	8.10	10.26	8.26	10.46	8.42	10.66
28.9	10.04	8.08	10.13	8.15	10.33	8.31	10.52	8.47	10.73
29.0	10.10	8.13	10.19	8.20	10.40	8.36	10.59	8.53	10.79
29.1	10.17	8.18	10.26	8.26	10.46	8.42	10.66	8.58	10.86
29.2	10.24	8.24	10.33	8.31	10.52	8.47	10.73	8.64	10.93
29.3	10.30	8.29	10.40	8.37	10.59	8.53	10.79	8.69	11.00
29.4	10.36	8.34	10.46	8.42	10.66	8.58	10.86	8.74	11.06
29.5	10.43	8.40	10.52	8.47	10.72	8.64	10.93	8.80	11.13
29.6	10.50	8.45	10.59	8.53	10.79	8.69	10.99	8.85	11.20
29.7	10.56	8.50	10.66	8.58	10.86	8.74	11.06	8.91	11.27
29.8	10.63	8.56	10.72	8.63	10.93	8.80	11.12	8.96	11.33
29.9	10.69	8.61	10.79	8.69	10.99	8.85	11.19	9.02	11.39
30.0	10.76	8.66	10.86	8.74	11.05	8.91	11.26	9.07	11.46
30.1	10.83	8.72	10.93	8.80	11.12	8.96	11.32	9.12	11.52
30.2	10.89	8.77	10.99	8.85	11.18	9.02	11.38	9.18	11.59
30.3	10.95	8.82	11.05	8.90	11.25	9.07	11.45	9.23	11.66
30.4	11.02	8.88	11.12	8.96	11.31	9.12	11.51	9.28	11.72
30.5	11.08	8.93	11.18	9.01	11.38	9.18	11.58	9.34	11.79
30.6	11.15	8.98	11.25	9.06	11.44	9.23	11.64	9.39	11.85
30.7	11.21	9.04	11.31	9.12	11.51	9.28	11.71	9.44	11.92
30.8	11.28	9.09	11.38	9.17	11.58	9.34	11.78	9.50	11.99
30.9	11.34	9.14	11.44	9.22	11.64	9.39	11.84	9.55	12.05
31.0	11.41	9.19	11.51	9.28	11.71	9.44	11.91	9.60	12.12
31.1	11.47	9.25	11.57	9.33	11.77	9.49	11.97	9.66	12.18
31.2	11.54	9.30	11.64	9.38	11.84	9.55	12.04	9.71	12.25
31.3	11.60	9.35	11.70	9.43	11.90	9.60	12.11	9.76	12.32
31.4	11.66	9.40	11.77	9.49	11.97	9.65	12.17	9.82	12.38
31.5	11.73	9.46	11.83	9.54	12.03	9.71	12.24	9.87	12.45
31.6	11.79	9.51	11.90	9.59	12.10	9.76	12.30	9.92	12.51
31.7	11.86	9.56	11.96	9.65	12.16	9.81	12.37	9.98	12.58
31.8	11.92	9.62	12.03	9.70	12.23	9.87	12.43	10.03	12.64
31.9	11.99	9.67	12.09	9.75	12.29	9.92	12.50	10.09	12.71
32.0	12.05	9.72	12.15	9.80	12.36	9.97	12.57	10.14	12.78
32.1	12.12	9.77	12.21	9.86	12.42	10.03	12.63	10.19	12.84
32.2	12.18	9.83	12.28	9.91	12.49	10.08	12.70	10.25	12.91
32.3	12.24	9.88	12.34	9.96	12.55	10.13	12.76	10.30	12.97
32.4	12.31	9.93	12.40	10.02	12.62	10.19	12.83	10.35	13.04
32.5	12.37	9.98	12.47	10.07	12.68	10.24	12.89	10.41	13.10
32.6	12.43	10.04	12.54	10.12	12.75	10.29	12.96	10.46	13.17
32.7	12.50	10.09	12.60	10.17	12.81	10.34	13.03	10.52	13.24
32.8	12.56	10.14	12.67	10.23	12.88	10.40	13.09	10.57	13.30
32.9	12.62	10.19	12.73	10.28	12.94	10.45	13.15	10.62	13.37

TABLE.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		SCALE READING
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	
8.14	10.31	*8.30*	10.51	*8.46*	10.72	*8.63*	10.93	*8.80*	28.0
8.19	10.38	*8.35*	10.58	*8.52*	10.79	*8.69*	10.99	*8.85*	28.1
8.25	10.45	*8.41*	10.65	*8.58*	10.86	*8.74*	11.06	*8.91*	28.2
8.30	10.52	*8.46*	10.72	*8.63*	10.93	*8.80*	11.13	*8.97*	28.3
8.36	10.59	*8.52*	10.79	*8.69*	11.00	*8.86*	11.20	*9.02*	28.4
8.41	10.66	*8.58*	10.86	*8.74*	11.06	*8.91*	11.27	*9.08*	28.5
8.47	10.72	*8.63*	10.93	*8.80*	11.13	*8.97*	11.33	*9.13*	28.6
8.52	10.79	*8.69*	11.00	*8.86*	11.20	*9.02*	11.40	*9.19*	28.7
8.58	10.86	*8.74*	11.06	*8.91*	11.27	*9.08*	11.47	*9.24*	28.8
8.64	10.93	*8.80*	11.13	*8.97*	11.33	*9.13*	11.54	*9.30*	28.9
8.69	11.00	*8.86*	11.20	*9.02*	11.40	*9.19*	11.61	*9.36*	29.0
8.75	11.06	*8.91*	11.27	*9.08*	11.47	*9.24*	11.68	*9.41*	29.1
8.80	11.13	*8.97*	11.33	*9.13*	11.54	*9.30*	11.75	*9.47*	27.2
8.86	11.20	*9.02*	11.40	*9.19*	11.60	*9.35*	11.81	*9.52*	29.3
8.91	11.27	*9.08*	11.47	*9.24*	11.67	*9.41*	11.88	*9.58*	29.4
8.97	11.33	*9.13*	11.54	*9.30*	11.74	*9.46*	11.94	*9.63*	29.5
9.02	11.39	*9.18*	11.60	*9.35*	11.81	*9.52*	12.01	*9.69*	29.6
9.08	11.46	*9.24*	11.67	*9.41*	11.87	*9.57*	12.08	*9.75*	29.7
9.13	11.53	*9.29*	11.74	*9.46*	11.94	*9.63*	12.15	*9.80*	29.8
9.18	11.60	*9.35*	11.81	*9.52*	12.01	*9.69*	12.22	*9.86*	29.9
9.24	11.66	*9.40*	11.87	*9.57*	12.08	*9.74*	12.29	*9.91*	30.0
9.29	11.73	*9.46*	11.93	*9.63*	12.14	*9.80*	12.36	*9.97*	30.1
9.34	11.79	*9.51*	12.00	*9.68*	12.21	*9.85*	12.42	*10.02*	30.2
9.40	11.86	*9.57*	12.07	*9.74*	12.28	*9.91*	12.49	*10.08*	30.3
9.45	11.93	*9.62*	12.13	*9.79*	12.34	*9.96*	12.56	*10.13*	30.4
9.50	11.99	*9.67*	12.20	*9.85*	12.41	*10.02*	12.63	*10.19*	30.5
9.56	12.06	*9.73*	12.27	*9.90*	12.48	*10.07*	12.70	*10.24*	30.6
9.61	12.13	*9.78*	12.34	*9.96*	12.55	*10.13*	12.77	*10.30*	30.7
9.67	12.19	*9.84*	12.40	*10.01*	12.61	*10.18*	12.84	*10.36*	30.8
9.72	12.26	*9.89*	12.47	*10.07*	12.68	*10.24*	12.90	*10.41*	30.9
9.77	12.32	*9.95*	12.54	*10.12*	12.75	*10.29*	12.97	*10.47*	31.0
9.83	12.39	*10.00*	12.60	*10.17*	12.82	*10.35*	13.04	*10.52*	31.1
9.88	12.46	*10.05*	12.67	*10.23*	12.89	*10.40*	13.11	*10.58*	31.2
9.94	12.52	*10.11*	12.74	*10.28*	12.95	*10.46*	13.17	*10.63*	31.3
9.99	12.59	*10.16*	12.81	*10.34*	13.02	*10.51*	13.24	*10.69*	31.4
10.04	12.66	*10.22*	12.87	*10.39*	13.09	*10.57*	13.31	*10.74*	31.5
10.10	12.72	*10.27*	12.94	*10.45*	13.15	*10.62*	13.37	*10.80*	31.6
10.15	12.79	*10.32*	13.01	*10.50*	13.22	*10.68*	13.44	*10.86*	31.7
10.21	12.85	*10.38*	13.07	*10.55*	13.29	*10.73*	13.51	*10.91*	31.8
10.26	12.92	*10.43*	13.14	*10.61*	13.35	*10.78*	13.57	*10.97*	31.9
10.31	12.99	*10.49*	13.20	*10.66*	13.42	*10.84*	13.64	*11.02*	32.0
10.37	13.05	*10.54*	13.27	*10.72*	13.49	*10.90*	13.71	*11.08*	32.1
10.42	13.12	*10.59*	13.34	*10.77*	13.55	*10.95*	13.77	*11.13*	32.2
10.48	13.18	*10.65*	13.40	*10.83*	13.62	*11.01*	13.84	*11.19*	32.3
10.53	13.25	*10.70*	13.47	*10.88*	13.69	*11.05*	13.91	*11.24*	32.4
10.58	13.32	*10.76*	13.53	*10.94*	13.75	*11.11*	13.97	*11.30*	32.5
10.64	13.38	*10.81*	13.60	*10.99*	13.82	*11.17*	14.04	*11.35*	32.6
10.69	13.45	*10.87*	13.66	*11.04*	13.89	*11.22*	14.11	*11.41*	32.7
10.75	13.51	*10.92*	13.73	*11.10*	13.95	*11.28*	14.17	*11.46*	32.8
10.80	13.58	*10.97*	13.80	*11.15*	14.02	*11.33*	14.24	*11.52*	32.9

8 ALCOHOL

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
33.0	12.69	*10.24*	12.79	*10.33*	13.01	*10.50*	13.22	*10.68*	13.43
33.1	12.76	*10.30*	12.86	*10.38*	13.07	*10.56*	13.28	*10.73*	13.50
33.2	12.82	*10.35*	12.92	*10.43*	13.13	*10.61*	13.35	*10.79*	13.56
33.3	12.88	*10.40*	12.99	*10.49*	13.20	*10.66*	13.41	*10.84*	13.63
33.4	12.95	*10.45*	13.05	*10.54*	13.26	*10.71*	13.48	*10.89*	13.69
33.5	13.01	*10.50*	13.11	*10.59*	13.32	*10.77*	13.54	*10.95*	13.76
33.6	13.08	*10.56*	13.18	*10.64*	13.39	*10.82*	13.61	*11.00*	13.82
33.7	13.14	*10.61*	13.24	*10.70*	13.45	*10.87*	13.67	*11.05*	13.89
33.8	13.20	*10.66*	13.30	*10.75*	13.52	*10.93*	13.74	*11.10*	13.95
33.9	13.26	*10.71*	13.37	*10.80*	13.58	*10.98*	13.80	*11.16*	14.02
34.0	13.33	*10.77*	13.43	*10.85*	13.64	*11.03*	13.86	*11.21*	14.08
34.1	13.39	*10.82*	13.49	*10.91*	13.71	*11.08*	13.93	*11.26*	14.15
34.2	13.45	*10.87*	13.56	*10.96*	13.77	*11.13*	13.99	*11.31*	14.21
34.3	13.52	*10.92*	13.62	*11.01*	13.83	*11.19*	14.06	*11.36*	14.27
34.4	13.58	*10.97*	13.68	*11.06*	13.90	*11.24*	14.12	*11.41*	14.34
34.5	13.64	*11.03*	13.75	*11.11*	13.96	*11.29*	14.18	*11.47*	14.40
34.6	13.70	*11.08*	13.81	*11.16*	14.02	*11.34*	14.25	*11.52*	14.47
34.7	13.77	*11.13*	13.87	*11.22*	14.08	*11.39*	14.31	*11.57*	14.53
34.8	13.83	*11.18*	13.94	*11.27*	14.14	*11.44*	14.37	*11.62*	14.59
34.9	13.89	*11.23*	14.00	*11.32*	14.20	*11.49*	14.43	*11.67*	14.66
35.0	13.96	*11.28*	14.06	*11.37*	14.27	*11.55*	14.50	*11.73*	14.72
35.1	14.02	*11.33*	14.13	*11.42*	14.33	*11.60*	14.56	*11.78*	14.78
35.2	14.08	*11.38*	14.19	*11.47*	14.39	*11.65*	14.62	*11.83*	14.85
35.3	14.14	*11.44*	14.25	*11.52*	14.46	*11.70*	14.69	*11.88*	14.91
35.4	14.21	*11.49*	14.31	*11.57*	14.52	*11.75*	14.75	*11.93*	14.97
35.5	14.27	*11.54*	14.38	*11.63*	14.59	*11.81*	14.81	*11.99*	15.04
35.6	14.33	*11.59*	14.44	*11.68*	14.65	*11.86*	14.87	*12.04*	15.10
35.7	14.39	*11.64*	14.50	*11.73*	14.71	*11.91*	14.94	*12.09*	15.16
35.8	14.46	*11.69*	14.56	*11.78*	14.78	*11.96*	15.00	*12.14*	15.23
35.9	14.52	*11.74*	14.63	*11.83*	14.84	*12.01*	15.06	*12.19*	15.29
36.0	14.58	*11.79*	14.69	*11.88*	14.90	*12.06*	15.13	*12.24*	15.35
36.1	14.64	*11.85*	14.75	*11.94*	14.97	*12.11*	15.19	*12.30*	15.42
36.2	14.71	*11.90*	14.81	*11.99*	15.03	*12.16*	15.25	*12.35*	15.48
36.3	14.77	*11.95*	14.88	*12.04*	15.09	*12.22*	15.32	*12.40*	15.54
36.4	14.83	*12.00*	14.94	*12.09*	15.16	*12.27*	15.38	*12.45*	15.61
36.5	14.89	*12.05*	15.00	*12.14*	15.22	*12.32*	15.44	*12.50*	15.67
36.6	14.96	*12.10*	15.06	*12.19*	15.28	*12.37*	15.51	*12.56*	15.73
36.7	15.02	*12.15*	15.13	*12.24*	15.35	*12.42*	15.57	*12.61*	15.80
36.8	15.08	*12.20*	15.19	*12.29*	15.41	*12.47*	15.63	*12.66*	15.86
36.9	15.14	*12.25*	15.25	*12.34*	15.47	*12.53*	15.70	*12.71*	15.92
37.0	15.20	*12.30*	15.31	*12.40*	15.53	*12.58*	15.76	*12.77*	15.99
37.1	15.27	*12.36*	15.38	*12.45*	15.60	*12.63*	15.82	*12.83*	16.05
37.2	15.33	*12.41*	15.44	*12.50*	15.66	*12.68*	15.89	*12.87*	16.11
37.3	15.39	*12.46*	15.50	*12.55*	15.72	*12.73*	15.95	*12.92*	16.18
37.4	15.45	*12.51*	15.56	*12.60*	15.79	*12.78*	16.01	*12.97*	16.24
37.5	15.51	*12.56*	15.63	*12.65*	15.85	*12.84*	16.08	*13.03*	16.30
37.6	15.57	*12.61*	15.69	*12.70*	15.91	*12.89*	16.14	*13.08*	16.37
37.7	15.64	*12.66*	15.75	*12.75*	15.97	*12.94*	16.20	*13.13*	16.43
37.8	15.70	*12.71*	15.81	*12.81*	16.04	*12.99*	16.26	*13.18*	16.49
37.9	15.76	*12.76*	15.88	*12.86*	16.10	*13.04*	16.33	*13.23*	16.56

TABLE.—Continued.

8

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
10.85	13.64	11.03	13.86	11.21	14.09	11.39	14.31	11.57	33.0
10.91	13.71	11.08	13.93	11.26	14.15	11.44	14.38	11.63	33.1
10.96	13.78	11.13	13.99	11.31	14.22	11.49	14.44	11.68	33.2
11.02	13.84	11.19	14.06	11.37	14.28	11.55	14.51	11.74	33.3
11.07	13.91	11.24	14.13	11.42	14.35	11.60	14.58	11.79	33.4
11.12	13.97	11.29	14.19	11.48	14.42	11.66	14.64	11.85	33.5
11.17	14.04	11.35	14.26	11.53	14.48	11.71	14.71	11.90	33.6
11.23	14.11	11.40	14.32	11.58	14.55	11.77	14.78	11.96	33.7
11.28	14.17	11.45	14.39	11.64	14.62	11.82	14.85	12.01	33.8
11.33	14.24	11.51	14.45	11.69	14.68	11.88	14.91	12.07	33.9
11.38	14.30	11.56	14.52	11.75	14.75	11.93	14.98	12.12	34.0
11.44	14.37	11.61	14.59	11.80	14.81	11.98	15.05	12.18	34.1
11.49	14.43	11.67	14.65	11.85	14.88	12.04	15.11	12.23	34.2
11.54	14.50	11.72	14.72	11.91	14.95	12.09	15.18	12.29	34.3
11.59	14.57	11.78	14.78	11.96	15.01	12.15	15.25	12.34	34.4
11.65	14.63	11.83	14.85	12.02	15.08	12.20	15.31	12.40	34.5
11.70	14.70	11.88	14.91	12.07	15.14	12.26	15.38	12.45	34.6
11.75	14.76	11.94	14.98	12.12	15.21	12.31	15.45	12.51	34.7
11.81	14.83	11.99	15.05	12.18	15.28	12.36	15.51	12.56	34.8
11.86	14.89	12.04	15.11	12.23	15.34	12.42	15.58	12.62	34.9
11.91	14.96	12.10	15.18	12.28	15.41	12.47	15.65	12.67	35.0
11.96	15.03	12.15	15.24	12.34	15.47	12.53	15.71	12.73	35.1
12.02	15.09	12.20	15.31	12.39	15.54	12.58	15.78	12.78	35.2
12.07	15.15	12.25	15.37	12.44	15.61	12.64	15.85	12.84	35.3
12.12	15.22	12.31	15.44	12.50	15.67	12.69	15.91	12.89	35.4
12.17	15.28	12.36	15.50	12.55	15.74	12.75	15.98	12.95	35.5
12.23	15.34	12.41	15.56	12.60	15.80	12.80	16.05	13.00	35.6
12.28	15.41	12.47	15.63	12.66	15.87	12.85	16.11	13.05	35.7
12.33	15.47	12.52	15.69	12.71	15.93	12.91	16.18	13.11	35.8
12.38	15.53	12.57	15.76	12.76	16.00	12.96	16.24	13.16	35.9
12.43	15.59	12.62	15.82	12.82	16.06	13.02	16.31	13.21	36.0
12.49	15.66	12.68	15.89	12.87	16.13	13.07	16.37	13.27	36.1
12.54	15.72	12.73	15.95	12.92	16.19	13.12	16.44	13.32	36.2
12.59	15.78	12.78	16.02	12.98	16.26	13.18	16.50	13.37	36.3
12.64	15.85	12.84	16.08	13.03	16.32	13.23	16.56	13.43	36.4
12.70	15.91	12.89	16.15	13.08	16.39	13.28	16.63	13.48	36.5
12.75	15.97	12.94	16.21	13.14	16.45	13.34	16.69	13.53	36.6
12.80	16.04	12.99	16.28	13.19	16.52	13.39	16.76	13.59	36.7
12.85	16.10	13.05	16.34	13.24	16.58	13.44	16.82	13.64	36.8
12.91	16.16	13.10	16.40	13.29	16.65	13.49	16.89	13.70	36.9
12.96	16.23	13.15	16.47	13.35	16.71	13.55	16.95	13.75	37.0
13.01	16.29	13.20	16.53	13.40	16.77	13.60	17.02	13.80	37.1
13.06	16.35	13.26	16.60	13.45	16.84	13.65	17.08	13.86	37.2
13.11	16.42	13.31	16.66	13.50	16.90	13.71	17.15	13.91	37.3
13.16	16.48	13.36	16.72	13.56	16.97	13.76	17.21	13.96	37.4
13.21	16.54	13.41	16.79	13.61	17.03	13.81	17.27	14.02	37.5
13.27	16.61	13.46	16.85	13.66	17.09	13.87	17.34	14.07	37.6
13.32	16.67	13.52	16.92	13.72	17.16	13.92	17.40	14.12	37.7
13.37	16.73	13.57	16.98	13.77	17.22	13.97	17.46	14.17	37.8
13.42	16.80	13.62	17.04	13.82	17.28	14.03	17.53	14.23	37.9

8 ALCOHOL

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
38.0	15.82	*12.81*	15.94	*12.91*	16.16	*13.09*	16.39	*13.28*	16.62
38.1	15.88	*12.86*	16.00	*12.96*	16.22	*13.14*	16.45	*13.33*	16.68
38.2	15.94	*12.91*	16.06	*13.01*	16.29	*13.19*	16.51	*13.38*	16.75
38.3	16.01	*12.96*	16.12	*13.06*	16.35	*13.25*	16.57	*13.44*	16.81
38.4	16.07	*13.02*	16.18	*13.11*	16.41	*13.30*	16.64	*13.49*	16.87
38.5	16.13	*13.07*	16.25	*13.16*	16.47	*13.35*	16.70	*13.54*	16.94
38.6	16.19	*13.12*	16.31	*13.21*	16.53	*13.40*	16.76	*13.59*	17.00
38.7	16.25	*13.17*	16.37	*13.26*	16.60	*13.45*	16.83	*13.64*	17.06
38.8	16.31	*13.22*	16.43	*13.31*	16.66	*13.50*	16.89	*13.69*	17.13
38.9	16.37	*13.27*	16.49	*13.37*	16.72	*13.55*	16.95	*13.74*	17.19
39.0	16.44	*13.32*	16.55	*13.42*	16.78	*13.61*	17.01	*13.79*	17.25
39.1	16.50	*13.37*	16.61	*13.47*	16.84	*13.66*	17.07	*13.85*	17.31
39.2	16.56	*13.42*	16.67	*13.52*	16.91	*13.71*	17.14	*13.90*	17.38
39.3	16.62	*13.47*	16.74	*13.57*	16.97	*13.76*	17.20	*13.95*	17.44
39.4	16.68	*13.52*	16.80	*13.62*	17.03	*13.81*	17.26	*14.00*	17.50
39.5	16.74	*13.57*	16.86	*13.67*	17.09	*13.86*	17.32	*14.05*	17.56
39.6	16.80	*13.62*	16.92	*13.72*	17.15	*13.91*	17.39	*14.10*	17.63
39.7	16.87	*13.68*	16.98	*13.77*	17.21	*13.96*	17.45	*14.15*	17.69
39.8	16.93	*13.73*	17.04	*13.82*	17.28	*14.02*	17.51	*14.21*	17.75
39.9	16.99	*13.78*	17.10	*13.87*	17.34	*14.07*	17.57	*14.26*	17.81
40.0	17.05	*13.83*	17.16	*13.92*	17.40	*14.12*	17.63	*14.31*	17.88
40.1	17.11	*13.88*	17.23	*13.98*	17.46	*14.17*	17.70	*14.36*	17.94
40.2	17.17	*13.93*	17.29	*14.03*	17.52	*14.22*	17.76	*14.41*	18.00
40.3	17.23	*13.98*	17.35	*14.08*	17.58	*14.27*	17.82	*14.46*	18.06
40.4	17.29	*14.03*	17.41	*14.13*	17.64	*14.32*	17.88	*14.51*	18.12
40.5	17.35	*14.08*	17.47	*14.18*	17.71	*14.37*	17.94	*14.56*	18.19
40.6	17.41	*14.13*	17.53	*14.23*	17.77	*14.42*	18.01	*14.62*	18.25
40.7	17.48	*14.18*	17.59	*14.28*	17.83	*14.47*	18.07	*14.67*	18.31
40.8	17.54	*14.23*	17.65	*14.33*	17.89	*14.52*	18.13	*14.72*	18.37
40.9	17.60	*14.28*	17.71	*14.38*	17.95	*14.57*	18.19	*14.77*	18.43
41.0	17.66	*14.33*	17.77	*14.43*	18.01	*14.62*	18.25	*14.82*	18.49
41.1	17.72	*14.38*	17.84	*14.48*	18.07	*14.68*	18.31	*14.87*	18.56
41.2	17.78	*14.43*	17.90	*14.53*	18.13	*14.73*	18.37	*14.92*	18.62
41.3	17.84	*14.48*	17.96	*14.58*	18.20	*14.78*	18.44	*14.97*	18.68
41.4	17.90	*14.53*	18.03	*14.63*	18.26	*14.83*	18.50	*15.03*	18.74
41.5	17.96	*14.58*	18.08	*14.68*	18.32	*14.88*	18.56	*15.08*	18.80
41.6	18.02	*14.63*	18.14	*14.73*	18.38	*14.93*	18.62	*15.13*	18.86
41.7	18.08	*14.68*	18.20	*14.78*	18.44	*14.98*	18.68	*15.18*	18.93
41.8	18.14	*14.73*	18.26	*14.83*	18.50	*15.03*	18.74	*15.23*	18.99
41.9	18.20	*14.78*	18.32	*14.88*	18.56	*15.08*	18.81	*15.28*	19.05
42.0	18.27	*14.83*	18.38	*14.93*	18.62	*15.13*	18.87	*15.33*	19.11
42.1	18.33	*14.88*	18.44	*14.98*	18.68	*15.18*	18.93	*15.38*	19.17
42.2	18.39	*14.93*	18.50	*15.03*	18.74	*15.23*	18.99	*15.43*	19.23
42.3	18.45	*14.98*	18.56	*15.08*	18.80	*15.28*	19.05	*15.48*	19.29
42.4	18.51	*15.03*	18.62	*15.13*	18.87	*15.33*	19.11	*15.53*	19.36
42.5	18.57	*15.08*	18.68	*15.18*	18.93	*15.38*	19.17	*15.58*	19.42
42.6	18.63	*15.13*	18.75	*15.23*	18.99	*15.43*	19.23	*15.63*	19.48
42.7	18.69	*15.18*	18.81	*15.28*	19.05	*15.48*	19.29	*15.69*	19.54
42.8	18.75	*15.23*	18.87	*15.33*	19.11	*15.53*	19.36	*15.74*	19.60
42.9	18.81	*15.28*	18.93	*15.38*	19.17	*15.58*	19.42	*15.79*	19.66

TABLE.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		SCALE READING
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	
13.47	16.86	13.67	17.11	13.87	17.35	14.08	17.59	14.28	38.0
13.53	16.92	13.72	17.17	13.93	17.41	14.13	17.66	14.33	38.1
13.58	16.99	13.78	17.23	13.98	17.47	14.18	17.72	14.38	38.2
13.63	17.05	13.83	17.30	14.03	17.54	14.23	17.78	14.43	38.3
13.68	17.11	13.88	17.36	14.08	17.60	14.29	17.85	14.49	38.4
13.73	17.18	13.93	17.42	14.14	17.66	14.34	17.91	14.54	38.5
13.79	17.24	13.98	17.48	14.19	17.73	14.39	17.97	14.59	38.6
13.84	17.30	14.04	17.55	14.24	17.79	14.44	18.04	14.64	38.7
13.89	17.36	14.09	17.61	14.29	17.85	14.49	18.10	14.70	38.8
13.94	17.43	14.14	17.67	14.34	17.92	14.55	18.16	14.75	38.9
13.99	17.49	14.19	17.74	14.40	17.98	14.60	18.23	14.80	39.0
14.05	17.55	14.24	17.80	14.45	18.04	14.65	18.29	14.85	39.1
14.10	17.62	14.30	17.86	14.50	18.11	14.70	18.35	14.91	39.2
14.15	17.68	14.35	17.92	14.55	18.17	14.76	18.42	14.96	39.3
14.20	17.74	14.40	17.99	14.60	18.23	14.81	18.48	15.01	39.4
14.25	17.81	14.45	18.05	14.66	18.30	14.86	18.54	15.06	39.5
14.30	17.87	14.50	18.11	14.71	18.36	14.91	18.61	15.12	39.6
14.36	17.93	14.56	18.18	14.76	18.42	14.96	18.67	15.17	39.7
14.41	17.99	14.61	18.24	14.81	18.48	15.02	18.73	15.22	39.8
14.46	18.06	14.66	18.30	14.87	18.55	15.07	18.80	15.27	39.9
14.51	18.12	14.71	18.36	14.92	18.61	15.12	18.86	15.32	40.0
14.56	18.18	14.77	18.43	14.97	18.67	15.17	18.92	15.38	40.1
14.61	18.24	14.82	18.49	15.02	18.74	15.22	18.99	15.43	40.2
14.67	18.30	14.87	18.55	15.07	18.80	15.27	19.05	15.48	40.3
14.72	18.37	14.92	18.61	15.12	18.86	15.33	19.11	15.53	40.4
14.77	18.43	14.97	18.68	15.17	18.92	15.38	19.18	15.59	40.5
14.82	18.49	15.03	18.74	15.23	18.99	15.43	19.24	15.64	40.6
14.87	18.55	15.08	18.80	15.28	19.05	15.48	19.30	15.69	40.7
14.92	18.61	15.13	18.86	15.33	19.11	15.53	19.37	15.74	40.8
14.97	18.68	15.18	18.93	15.38	19.18	15.59	19.43	15.80	40.9
15.03	18.74	15.23	18.99	15.43	19.24	15.64	19.49	15.85	41.0
15.08	18.80	15.28	19.05	15.48	19.30	15.69	19.56	15.90	41.1
15.13	18.86	15.33	19.11	15.53	19.36	15.74	19.62	15.95	41.2
15.18	18.93	15.38	19.17	15.58	19.43	15.79	19.68	16.01	41.3
15.23	18.99	15.43	19.24	15.64	19.49	15.84	19.75	16.06	41.4
15.28	19.05	15.48	19.30	15.69	19.55	15.90	19.81	16.11	41.5
15.33	19.11	15.53	19.36	15.74	19.61	15.95	19.87	16.16	41.6
15.38	19.17	15.58	19.42	15.79	19.68	16.00	19.94	16.21	41.7
15.43	19.23	15.63	19.48	15.84	19.74	16.05	20.00	16.27	41.8
15.48	19.29	15.69	19.55	15.89	19.80	16.10	20.06	16.32	41.9
15.53	19.36	15.74	19.61	15.94	19.86	16.16	20.13	16.37	42.0
15.58	19.42	15.79	19.67	15.99	19.93	16.21	20.19	16.42	42.1
15.63	19.48	15.84	19.73	16.05	19.99	16.26	20.25	16.48	42.2
15.69	19.54	15.89	19.80	16.10	20.05	16.31	20.31	16.53	42.3
15.74	19.60	15.94	19.86	16.15	20.11	16.36	20.38	16.58	42.4
15.79	19.66	15.99	19.92	16.20	20.18	16.41	20.44	16.63	42.5
15.84	19.72	16.04	19.98	16.25	20.24	16.47	20.50	16.69	52.6
15.89	19.79	16.09	20.04	16.30	20.30	16.52	20.57	16.74	42.7
15.94	19.85	16.14	20.10	16.35	20.36	16.57	20.63	16.79	42.8
15.99	19.91	16.19	20.17	16.41	20.43	16.62	20.69	16.84	42.9

8 ALCOHOL

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
43.0	**18.87**	*15.33*	**18.99**	*15.43*	**19.23**	*15.63*	**19.48**	*15.84*	**19.72**
43.1	**18.93**	*15.38*	**19.05**	*15.48*	**19.29**	*15.68*	**19.54**	*15.89*	**19.79**
43.2	**18.99**	*15.43*	**19.11**	*15.53*	**19.35**	*15.73*	**19.60**	*15.94*	**19.85**
43.3	**19.05**	*15.48*	**19.17**	*15.58*	**19.41**	*15.78*	**19.66**	*15.99*	**19.91**
43.4	**19.11**	*15.53*	**19.23**	*15.63*	**19.47**	*15.83*	**19.72**	*16.04*	**19.97**
43.5	**19.17**	*15.58*	**19.29**	*15.68*	**19.53**	*15.88*	**19.79**	*16.09*	**20.03**
43.6	**19.23**	*15.63*	**19.35**	*15.73*	**19.59**	*15.93*	**19.85**	*16.14*	**20.09**
43.7	**19.29**	*15.68*	**19.41**	*15.78*	**19.65**	*15.98*	**19.91**	*16.19*	**20.15**
43.8	**19.35**	*15.73*	**19.47**	*15.83*	**19.72**	*16.03*	**19.97**	*16.24*	**20.21**
43.9	**19.40**	*15.78*	**19.53**	*15.88*	**19.78**	*16.08*	**20.03**	*16.29*	**20.27**
44.0	**19.46**	*15.83*	**19.59**	*15.93*	**19.84**	*16.13*	**20.09**	*16.34*	**20.34**
44.1	**19.52**	*15.88*	**19.65**	*15.98*	**19.90**	*16.18*	**20.15**	*16.39*	**20.40**
44.2	**19.58**	*15.93*	**19.71**	*16.03*	**19.96**	*16.23*	**20.21**	*16.44*	**20.46**
44.3	**19.64**	*15.98*	**19.77**	*16.08*	**20.02**	*16.28*	**20.27**	*16.49*	**20.52**
44.4	**19.70**	*16.02*	**19.83**	*16.13*	**20.08**	*16.33*	**20.33**	*16.55*	**20.58**
44.5	**19.76**	*16.07*	**19.89**	*16.18*	**20.14**	*16.38*	**20.39**	*16.60*	**20.64**
44.6	**19.82**	*16.12*	**19.95**	*16.23*	**20.20**	*16.43*	**20.45**	*16.65*	**20.70**
44.7	**19.88**	*16.17*	**20.01**	*16.27*	**20.26**	*16.48*	**20.52**	*16,70*	**20.76**
44.8	**19.94**	*16.22*	**20.07**	*16.32*	**20.32**	*16.53*	**20.58**	*16.75*	**20.82**
44.9	**20.00**	*16.27*	**20.12**	*16.37*	**20.38**	*16.58*	**20.64**	*16.80*	**20.88**
45.0	**20.06**	*16.32*	**20.18**	*16.42*	**20.44**	*16.63*	**20.70**	*16.85*	**20.95**
45.1	**20.12**	*16.37*	**20.24**	*16.47*	**20.50**	*16.68*	**20.76**	*16.90*	**21.01**
45.2	**20.18**	*16.41*	**20.30**	*16.52*	**20.56**	*16.73*	**20.82**	*16.95*	**21.07**
45.3	**20.24**	*16.46*	**20.36**	*16.57*	**20.62**	*16.78*	**20.88**	*17.00*	**21.13**
45.4	**20.29**	*16.51*	**20.42**	*16.62*	**20.68**	*18.83*	**20.94**	*17.05*	**21.19**
45.5	**20.35**	*16.56*	**20.48**	*16.67*	**20.74**	*16.88*	**21.00**	*17.10*	**21.25**
45.6	**20.41**	*16.61*	**20.54**	*16.72*	**20.80**	*16.93*	**21.06**	*17.15*	**21.31**
45.7	**20.47**	*16.66*	**20.60**	*16.76*	**20.86**	*16.98*	**21.12**	*17.20*	**21.37**
45.8	**20.53**	*16.71*	**20.66**	*16.81*	**20.92**	*17.03*	**21.18**	*17.25*	**21.43**
45.9	**20.59**	*16.76*	**20.72**	*16.86*	**20.98**	*17.08*	**21.24**	*17.30*	**21.49**
46.0	**20.65**	*16.80*	**20.78**	*16.91*	**21.04**	*17.13*	**21.30**	*17.35*	**21.54**
46.1	**20.71**	*16.85*	**20.83**	*16.96*	**21.10**	*17.18*	**21.36**	*17.40*	**21.60**
46.2	**20.76**	*16.90*	**20.89**	*17.01*	**21.16**	*17.23*	**21.42**	*17.45*	**21.66**
46.3	**20.82**	*16.95*	**20.95**	*17.06*	**21.22**	*17.28*	**21.48**	*17.50*	**21.72**
46.4	**20.88**	*17.00*	**21.01**	*17.11*	**21.28**	*17.33*	**21.54**	*17.55*	**21.78**
46.5	**20.94**	*17.05*	**21.07**	*17.16*	**21.34**	*17.38*	**21.60**	*17.60*	**21.84**
46.6	**21.00**	*17.10*	**21.13**	*17.21*	**21.40**	*17.43*	**21.66**	*17.65*	**21.90**
46.7	**21.06**	*17.15*	**21.19**	*17.26*	**21.46**	*17.48*	**21.72**	*17.70*	**21.96**
46.8	**21.12**	*17.20*	**21.25**	*17.31*	**21.52**	*17.53*	**21.78**	*17.75*	**22.02**
46.9	**21.18**	*17.25*	**21.31**	*17.36*	**21.58**	*17.58*	**21.84**	*17.80*	**22.09**
47.0	**21.24**	*17.30*	**21.37**	*17.41*	**21.64**	*17.63*	**21.90**	*17.85*	**22.15**
47.1	**21.30**	*17.35*	**21.43**	*17.46*	**21.70**	*17.68*	**21.96**	*17.90*	**22.21**
47.2	**21.36**	*17.40*	**21.49**	*17.51*	**21.76**	*17.73*	**22.02**	*17.95*	**22.27**
47.3	**21.42**	*17.45*	**21.55**	*17.56*	**21.82**	*17.78*	**22.08**	*18.01*	**22.33**
47.4	**21.48**	*17.50*	**21.61**	*17.61*	**21.88**	*17.83*	**22.15**	*18.06*	**22.39**
47.5	**21.54**	*17.55*	**21.67**	*17.66*	**21.94**	*17.88*	**22.21**	*18.11*	**22.45**
47.6	**21.60**	*17.60*	**21.73**	*17.71*	**22.00**	*17.94*	**22.27**	*18.16*	**22.51**
47.7	**21.66**	*17.65*	**21.79**	*17.76*	**22.06**	*17.99*	**22.33**	*18.21*	**22.58**
47.8	**21.72**	*17.70*	**21.85**	*17.81*	**22.12**	*18.04*	**22.39**	*18.26*	**22.64**
47.9	**21.78**	*17.75*	**21.91**	*17.86*	**22.18**	*18.09*	**22.45**	*18.31*	**22.70**

TABLE.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
16.04	19.97	16.24	20.23	16.46	20.49	16.67	20.75	16.90	43.0
16.09	20.03	16.30	20.29	16.51	20.55	16.73	20.82	16.95	43.1
16.14	20.09	16.35	20.35	16.56	20.61	16.78	20.88	17.00	43.2
16.19	20.15	16.40	20.41	16.61	20.68	16.83	20.94	17.05	43.3
16.24	20.21	16.45	20.47	16.66	20.74	16.88	21.01	17.10	43.4
16.29	20.28	16.50	20.53	16.71	20.80	16.93	21.07	17.16	43.5
16.34	20.34	16.55	20.60	16.76	20.86	16.98	21.13	17.21	43.6
16.40	20.40	16.60	20.66	16.82	20.93	17.04	21.19	17.26	43.7
16.45	20.46	16.65	20.72	16.87	20.99	17.09	21.25	17.31	43.8
16.50	20.52	16.70	20.78	16.92	21.05	17.14	21.32	17.36	43.9
16.55	20.58	16.76	20.84	16.97	21.11	17.19	21.38	17.41	44.0
16.60	20.64	16.81	20.90	17.02	21.17	17.24	21.44	17.47	44.1
16.65	20.71	16.86	20.96	17.07	21.23	17.29	21.50	17.52	44.2
16.70	20.77	16.91	21.02	17.12	21.30	17.35	21.57	17.57	44.3
16.75	20.83	16.96	21.09	17.17	21.36	17.40	21.63	17.62	44.4
16.80	20.89	17.01	21.15	17.22	21.42	17.45	21.69	17.67	44.5
16.85	20.95	17.06	21.21	17.28	21.48	17.50	21.75	17.73	44.6
16.90	21.01	17.11	21.27	17.33	21.54	17.55	21.81	17.78	44.7
16.95	21.07	17.16	21.33	17.38	21.60	17.60	21.88	17.83	44.8
17.01	21.13	17.21	21.39	17.43	21.67	17.65	21.94	17.88	44.9
17.06	21.19	17.26	21.45	17.48	21.73	17.71	22.00	17.93	45.0
17.11	21.25	17.31	21.52	17.53	21.79	17.76	22.06	17.98	45.1
17.16	21.31	17.36	21.58	17.58	21.85	17.81	22.13	18.04	45.2
17.21	21.37	17.41	21.64	17.63	21.91	17.86	22.19	18.09	45.3
17.26	21.43	17.46	21.70	17.68	21.98	17.91	22.25	18.14	45.4
17.31	21.49	17.51	21.76	17.73	22.04	17.96	22.32	18.20	45.5
17.36	21.55	17.56	21.82	17.79	22.10	18.02	22.38	18.25	45.6
17.41	21.61	17.61	21.88	17.84	22.16	18.07	22.45	18.30	45.7
17.46	21.67	17.66	21.94	17.89	22.23	18.12	22.51	18.36	45.8
17.51	21.73	17.71	22.01	17.94	22.29	18.17	22.57	18.41	45.9
17.56	21.79	17.76	22.07	17.99	22.35	18.23	22.64	18.47	46.0
17.61	21.85	17.81	22.13	18.04	22.42	18.28	22.70	18.52	46.1
17.66	21.91	17.86	22.19	18.09	22.48	18.33	22.76	18.57	46.2
17.71	21.97	17.91	22.26	18.15	22.54	18.39	22.83	18.63	46.3
17.76	22.03	17.96	22.32	18.20	22.61	18.44	22.89	18.68	46.4
17.81	22.09	18.01	22.38	18.25	22.67	18.49	22.96	18.73	46.5
17.86	22.16	18.06	22.44	18.30	22.73	18.55	23.02	18.79	46.6
17.91	22.22	18.11	22.51	18.36	22.80	18.60	23.08	18.84	46.7
17.96	22.28	18.17	22.57	18.41	22.86	18.65	23.15	18.90	46.8
18.01	22.34	18.22	22.63	18.46	22.92	18.70	23.21	18.95	46.9
18.06	22.41	18.27	22.69	18.51	22.99	18.76	23.28	19.00	47.0
18.11	22.47	18.32	22.76	18.57	23.05	18.81	23.34	19.05	47.1
18.16	22.53	18.38	22.82	18.62	23.12	18.86	23.41	19.11	47.2
18.21	22.59	18.43	22.88	18.67	23.18	18.92	23.47	19.16	47.3
18.26	22.66	18.48	22.94	18.72	23.24	18.97	23.54	19.22	47.4
18.31	22.72	18.53	23.01	18.78	23.31	19.02	23.60	19.27	47.5
18.36	22.78	18.58	23.07	18.83	23.37	19.08	23.67	19.33	47.6
18.42	22.84	18.64	23.13	18.88	23.44	19.13	23.73	19.38	47.7
18.47	22.91	18.69	23.20	18.93	23.50	19.18	23.80	19.43	47.8
18.52	22.97	18.74	23.26	18.99	23.56	19.24	23.86	19.49	47.9

8 ALCOHOL

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
48.0	21.84	*17.80*	21.97	*17.91*	22.24	*18.14*	22.51	*18.36*	22.76
48.1	21.90	*17.85*	22.03	*17.96*	22.30	*18.19*	22.57	*18.41*	22.82
48.2	21.96	*17.90*	22.09	*18.01*	22.36	*18.24*	22.63	*18.46*	22.88
48.3	22.02	*17.95*	22.15	*18.06*	22.42	*18.29*	22.69	*18.51*	22.94
48.4	22.08	*18.00*	22.21	*18.11*	22.48	*18.34*	22.75	*18.56*	23.01
48.5	22.14	*18.05*	22.27	*18.16*	22.54	*18.39*	22.81	*18.61*	23.07
48.6	22.20	*18.10*	22.33	*18.21*	22.60	*18.44*	22.87	*18.66*	23.13
48.7	22.26	*18.15*	22.39	*18.26*	22.66	*18.49*	22.93	*18.71*	23.19
48.8	22.32	*18.20*	22.45	*18.31*	22.72	*18.54*	22.99	*18.76*	23.26
48.9	22.38	*18.25*	22.51	*18.36*	22.78	*18.59*	23.06	*18.81*	23.32
49.0	22.44	*18.30*	22.57	*18.41*	22.84	*18.64*	23.12	*18.86*	23.38
49.1	22.50	*18.35*	22.63	*18.46*	22.90	*18.69*	23.18	*18.91*	23.44
49.2	22.56	*18.40*	22.69	*18.51*	22.96	*18.74*	23.24	*18.96*	23.51
49.3	22.62	*18.45*	22.75	*18.56*	23.02	*18.79*	23.30	*19.02*	23.57
49.4	22.68	*18.50*	22.81	*18.61*	23.08	*18.84*	23.36	*19.07*	23.63
49.5	22.74	*18.55*	22.87	*18.66*	23.15	*18.89*	23.42	*19.12*	23.69
49.6	22.80	*18.60*	22.93	*18.71*	23.21	*18.94*	23.48	*19.17*	23.76
49.7	22.86	*18.65*	22.99	*18.76*	23.27	*18.99*	23.55	*19.22*	23.82
49.8	22.92	*18.70*	23.05	*18.81*	23.33	*19.04*	23.61	*19.27*	23.88
49.9	22.98	*18.75*	23.11	*18.86*	23.39	*19.09*	23.67	*19.32*	23.94
50.0	23.04	*18.80*	23.17	*18.91*	23.45	*19.14*	23.73	*19.38*	24.01
50.1	23.10	*18.85*	23.23	*18.96*	23.51	*19.19*	23.79	*19.43*	24.07
50.2	23.16	*18.90*	23.30	*19.02*	23.57	*19.24*	23.85	*19.48*	24.13
50.3	23.22	*18.95*	23.36	*19.07*	23.63	*19.29*	23.91	*19.53*	24.19
50.4	23.28	*19.00*	23.42	*19.12*	23.69	*19.35*	23.98	*19.58*	24.26
50.5	23.34	*19.05*	23.48	*19.17*	23.75	*19.40*	24.04	*19.63*	24.32
50.6	23.40	*19.10*	23.54	*19.22*	23.81	*19.45*	24.10	*19.69*	24.38
50.7	23.46	*19.15*	23.60	*19.27*	23.87	*19.50*	24.16	*19.74*	24.45
50.8	23.51	*19.20*	23.66	*19.32*	23.93	*19.55*	24.22	*19.79*	24.51
50.9	23.57	*19.25*	23.72	*19.37*	23.99	*19.60*	24.28	*19.84*	24.57
51.0	23.63	*19.30*	23.78	*19.42*	24.05	*19.65*	24.35	*19.89*	24.64
51.1	23.69	*19.35*	23.84	*19.47*	24.12	*19.70*	24.41	*19.94*	24.70
51.2	23.75	*19.40*	23.90	*19.52*	24.18	*19.75*	24.47	*20.00*	24.76
51.3	23.81	*19.45*	23.96	*19.57*	24.24	*19.80*	24.53	*20.05*	24.82
51.4	23.87	*19.50*	24.02	*19.62*	24.30	*19.85*	24.59	*20.10*	24.89
51.5	23.93	*19.55*	24.08	*19.67*	24.36	*19.90*	24.65	*20.15*	24.95
51.6	23.99	*19.60*	24.14	*19.72*	24.42	*19.95*	24.72	*20.20*	25.01
51.7	24.05	*19.65*	24.20	*19.77*	24.48	*20.01*	24.78	*20.26*	25.07
51.8	24.11	*19.70*	24.26	*19.82*	24.54	*20.06*	24.84	*20.31*	25.14
51.9	24.17	*19.75*	24.32	*19.87*	24.60	*20.11*	24.90	*20.36*	25.20
52.0	24.23	*19.80*	24.38	*19.92*	24.66	*20.16*	24.96	*20.41*	25.27
52.1	24.30	*19.85*	24.44	*19.97*	24.73	*20.21*	25.03	*20.46*	25.33
52.2	24.36	*19.90*	24.50	*20.02*	24.79	*20.26*	25.09	*20.52*	25.39
52.3	24.42	*19.95*	24.56	*20.07*	24.85	*20.31*	25.15	*20.57*	25.46
52.4	24.48	*20.00*	24.62	*20.12*	24.91	*20.37*	25.21	*20.62*	25.52
52.5	24.54	*20.05*	24.68	*20.17*	24.97	*20.42*	25.28	*20.67*	25.58
52.6	24.60	*20.10*	24.74	*20.22*	25.03	*20.47*	25.34	*20.72*	25.65
52.7	24.66	*20.15*	24.80	*20.28*	25.09	*20.52*	25.40	*20.78*	25.71
52.8	24.72	*20.20*	24.86	*20.33*	25.15	*20.57*	25.46	*20.83*	25.77
52.9	24.78	*20.25*	24.92	*20.38*	25.22	*20.62*	25.53	*20.88*	25.84

TABLE.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
18.57	23.03	18.79	23.32	19.04	23.63	19.29	23.93	19.54	48.0
18.62	23.09	18.84	23.39	19.09	23.69	19.34	23.99	19.60	48.1
18.67	23.16	18.90	23.45	19.14	23.76	19.40	24.06	19.65	48.2
18.72	23.22	18.95	23.52	19.20	23.83	19.45	24.13	19.71	48.3
18.77	23.28	19.00	23.58	19.25	23.89	19.50	24.19	19.76	48.4
18.83	23.35	19.06	23.64	19.31	23.96	19.56	24.26	19.82	48.5
18.88	23.41	19.11	23.71	19.36	24.02	19.61	24.32	19.87	48.6
18.93	23.47	19.16	23.77	19.41	24.08	19.67	24.39	19.92	48.7
18.98	23.54	19.21	23.83	19.47	24.14	19.72	24.45	19.98	48.8
19.03	23.60	19.27	23.90	19.52	24.21	19.77	24.52	20.03	48.9
19.08	23.66	19.32	23.96	19.57	24.27	19.83	24.59	20.09	49.0
19.14	23.73	19.37	24.03	19.63	24.34	19.88	24.65	20.15	49.1
19.19	23.79	19.43	24.09	19.68	24.40	19.94	24.72	20.20	49.2
19.24	23.85	19.48	24.15	19.73	24.47	19.99	24.78	20.26	49.3
19.29	23.92	19.54	24.22	19.79	24.53	20.04	24.85	20.31	49.4
19.35	23.98	19.59	24.28	19.84	24.60	20.10	24.91	20.37	49.5
19.40	24.04	19.64	24.35	19.89	24.66	20.15	24.98	20.42	49.6
19.45	24.11	19.70	24.41	19.95	24.73	20.21	25.05	20.48	49.7
19.51	24.17	19.75	24.48	20.00	24.79	20.27	25.11	20.54	49.8
19.56	24.24	19.80	24.54	20.05	24.86	20.32	25.18	20.59	49.9
19.61	24.30	19.86	24.61	20.11	24.92	20.38	25.25	20.65	50.0
19.66	24.37	19.91	24.67	20.16	24.99	20.43	25.31	20.70	50.1
19.72	24.43	19.96	24.74	20.22	25.05	20.49	25.38	20.76	50.2
19.77	24.49	20.02	24.80	20.27	25.12	20.54	25.45	20.82	50.3
19.82	24.56	20.07	24.86	20.33	25.18	20.60	25.51	20.87	50.4
19.87	24.62	20.12	24.93	20.38	25.25	20.65	25.58	20.93	50.5
19.93	24.69	20.18	24.99	20.44	25.32	20.71	25.65	20.98	50.6
19.98	24.75	20.23	25.06	20.49	25.38	20.76	25.71	21.04	50.7
20.03	24.81	20.29	25.12	20.55	25.45	20.82	25.78	21.10	50.8
20.08	24.88	20.34	25.19	20.60	25.51	20.87	25.85	21.15	50.9
20.14	24.94	20.39	25.25	20.66	25.58	20.93	25.91	21.21	51.0
20.19	25.01	20.45	25.32	20.71	25.64	20.98	25.98	21.27	51.1
20.24	25.07	20.50	25.38	20.77	25.71	21.04	26.05	21.32	51.2
20.30	25.13	20.55	25.45	20.82	25.78	21.09	26.11	21.38	51.3
20.35	25.20	20.61	25.51	20.87	25.84	21.15	26.18	21.44	51.4
20.40	25.26	20.66	25.58	20.93	25.91	21.21	26.25	21.49	51.5
20.46	25.33	20.72	25.64	20.98	25.97	21.26	26.32	21.55	51.6
20.51	25.39	20.77	25.71	21.04	26.04	21.32	26.39	21.61	51.7
20.56	25.46	20.82	25.77	21.09	26.11	21.37	26.45	21.66	51.8
20.61	25.52	20.88	25.84	21.15	26.17	21.43	26.52	21.72	51.9
20.67	25.58	20.93	25.90	21.20	26.24	21.49	26.59	21.78	52.0
20.72	25.65	20.98	25.97	21.26	26.31	21.54	26.66	21.83	52.1
20.77	25.71	21.04	26.03	21.31	26.37	21.60	26.72	21.89	52.2
20.83	25.78	21.09	26.10	21.37	26.44	21.65	26.79	21.95	52.3
20.88	25.84	21.15	26.16	21.42	26.51	21.71	26.86	22.01	52.4
20.93	25.90	21.20	26.23	21.48	26.57	21.77	26.93	22.06	52.5
20.98	25.97	21.26	26.29	21.53	26.64	21.82	26.99	22.12	52.6
21.04	26.03	21.31	26.36	21.59	26.71	21.88	27.06	22.18	52.7
21.09	26.10	21.36	26.42	21.64	26.77	21.93	27.13	22.24	52.8
21.15	26.16	21.42	26.49	21.70	26.84	21.99	27.20	22.29	52.9

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
53.0	24.84	20.30	24.98	20.43	25.28	20.68	25.59	20.93	25.90
53.1	24.90	20.36	25.04	20.48	25.34	20.73	25.65	20.98	25.96
53.2	24.96	20.41	25.10	20.53	25.40	20.78	25.71	21.04	26.03
53.3	25.02	20.46	25.16	20.58	25.46	20.83	25.77	21.09	26.09
53.4	25.08	20.51	25.23	20.63	25.52	20.88	25.84	21.14	26.15
53.5	25.14	20.56	25.29	20.68	25.59	20.93	25.90	21.20	26.22
53.6	25.20	20.61	25.35	20.74	25.65	20.98	25.96	21.25	26.28
53.7	25.26	20.66	25.41	20.79	25.71	21.04	26.03	21.30	26.35
53.8	25.32	20.71	25.47	20.84	25.77	21.09	26.09	21.36	26.41
53.9	25.38	20.76	25.53	20.89	25.83	21.14	26.15	21.41	26.47
54.0	25.44	20.81	25.59	20.94	25.90	21.19	26.22	21.47	26.54
54.1	25.50	20.86	25.65	20.99	25.96	21.25	26.28	21.52	26.60
54.2	25.56	20.91	25.71	21.04	26.02	21.30	26.34	21.57	26.67
54.3	25.62	20.96	25.77	21.09	26.08	21.35	26.41	21.63	26.73
54.4	25.68	21.02	25.84	21.14	26.14	21.40	26.47	21.68	26.79
54.5	25.75	21.07	25.90	21.20	26.20	21.46	26.53	21.73	26.86
54.6	25.81	21.12	25.96	21.25	26.27	21.51	26.59	21.79	26.92
54.7	25.87	21.17	26.02	21.30	26.33	21.56	26.66	21.84	26.99
54.8	25.93	21.22	26.08	21.35	26.39	21.62	26.72	21.90	27.05
54.9	25.99	21.27	26.14	21.40	26.45	21.67	26.78	21.95	27.11
55.0	26.05	21.32	26.20	21.45	26.52	21.72	26.85	22.00	27.18
55.1	26.11	21.37	26.26	21.51	26.58	21.77	26.91	22.05	27.24
55.2	26.17	21.43	26.32	21.56	26.64	21.83	26.97	22.11	27.31
55.3	26.23	21.48	26.38	21.61	26.70	21.88	27.04	22.16	27.37
55.4	26.29	21.53	26.45	21.66	26.76	21.93	27.10	22.21	27.43
55.5	26.35	21.58	26.51	21.71	26.83	21.98	27.16	22.26	27.49
55.6	26.41	21.63	26.57	21.76	26.89	22.04	27.23	22.32	27.55
55.7	26.47	21.68	26.63	21.81	26.95	22.09	27.29	22.37	27.62
55.8	26.53	21.73	26.69	21.87	27.01	22.14	27.35	22.42	27.69
55.9	26.59	21.79	26.75	21.92	27.07	22.19	27.41	22.48	27.75
56.0	26.65	21.84	26.81	21.97	27.14	22.24	27.48	22.53	27.82
56.1	26.72	21.89	26.87	22.02	27.20	22.30	27.54	22.58	27.88
56.2	26.78	21.94	26.93	22.07	27.26	22.35	27.60	22.64	27.94
56.3	26.84	21.99	26.99	22.12	27.32	22.40	27.66	22.69	28.01
56.4	26.90	22.04	27.05	22.18	27.38	22.45	27.73	22.74	28.07
56.5	26.96	22.09	27.12	22.23	27.44	22.50	27.79	22.79	28.14
56.6	27.02	22.14	27.18	22.28	27.51	22.56	27.85	22.85	28.20
56.7	27.08	22.19	27.24	22.33	27.57	22.61	27.91	22.90	28.26
56.8	27.14	22.25	27.30	22.38	27.63	22.66	27.98	22.95	28.33
56.9	27.20	22.30	27.36	22.43	27.69	22.71	28.04	23.01	28.39
57.0	27.26	22.35	27.42	22.48	27.75	22.77	28.10	23.06	28.46
57.1	27.32	22.40	27.48	22.54	27.81	22.82	28.16	23.11	28.52
57.2	27.38	22.45	27.54	22.59	27.88	22.87	28.23	23.17	28.59
57.3	27.44	22.50	27.60	22.64	27.94	22.92	28.29	23.22	28.65
57.4	27.50	22.55	27.66	22.69	28.00	22.97	28.35	23.27	28.72
57.5	27.56	22.61	27.73	22.74	28.06	23.03	28.42	23.33	28.78
57.6	27.62	22.66	27.79	22.79	28.13	23.08	28.48	23.38	28.85
57.7	27.68	22.71	27.85	22.85	28.19	23.13	28.54	23.43	28.91
57.8	27.75	22.76	27.91	22.90	28.25	23.19	28.60	23.49	28.97
57.9	27.81	22.81	27.97	22.95	28.31	23.24	28.67	23.54	29.04

TABLE.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
21.20	26.23	*21.47*	26.56	*21.75*	26.91	*22.05*	27.27	*22.35*	53.0
21.25	26.29	*21.53*	26.62	*21.81*	26.97	*22.10*	27.33	*22.41*	53.1
21.31	26.35	*21.58*	26.69	*21.86*	27.04	*22.16*	27.40	*22.47*	53.2
21.36	26.42	*21.64*	26.75	*21.92*	27.11	*22.22*	27.47	*22.52*	53.3
21.42	26.48	*21.69*	26.82	*21.97*	27.17	*22.27*	27.54	*22.58*	53.4
21.47	26.55	*21.74*	26.88	*22.03*	27.24	*22.33*	27.61	*22.64*	53.5
21.52	26.61	*21.80*	26.95	*22.08*	27.31	*22.39*	27.67	*22.70*	53.6
21.58	26.68	*21.85*	27.01	*22.14*	27.38	*22.44*	27.74	*22.75*	53.7
21.63	26.74	*21.91*	27.08	*22.20*	27.44	*22.50*	27.81	*22.81*	53.8
21.69	26.81	*21.96*	27.15	*22.25*	27.51	*22.56*	27.88	*22.87*	53.9
21.74	26.87	*22.02*	27.21	*22.31*	27.58	*22.61*	27.95	*22.93*	54.0
21.79	26.94	*22.07*	27.28	*22.37*	27.64	*22.67*	28.01	*22.98*	54.1
21.85	27.00	*22.13*	27.35	*22.42*	27.71	*22.73*	28.08	*23.04*	54.2
21.90	27.07	*22.18*	27.41	*22.48*	27.78	*22.78*	28.15	*23.10*	54.3
21.96	27.13	*22.24*	27.48	*22.53*	27.85	*22.84*	28.22	*23.16*	54.4
22.01	27.20	*22.29*	27.55	*22.59*	27.91	*22.90*	28.29	*23.22*	54.5
22.06	27.26	*22.35*	27.61	*22.65*	27.98	*22.95*	28.36	*23.27*	54.6
22.12	27.33	*22.40*	27.68	*22.70*	28.05	*23.01*	28.43	*23.33*	54.7
22.17	27.39	*22.46*	27.75	*22.76*	28.11	*23.07*	28.49	*23.39*	54.8
22.23	27.46	*22.51*	27.81	*22.81*	28.18	*23.13*	28.56	*23.45*	54.9
22.28	27.52	*22.57*	27.88	*22.87*	28.25	*23.18*	28.63	*23.51*	55.0
22.33	27.59	*22.63*	27.95	*22.93*	28.32	*23.24*	28.70	*23.56*	55.1
22.39	27.65	*22.68*	28.01	*22.98*	28.38	*23.30*	28.77	*23.62*	55.2
22.44	27.72	*22.74*	28.08	*23.04*	28.45	*23.35*	28.84	*23.68*	55.3
22.49	27.78	*22.79*	28.15	*23.10*	28.52	*23.41*	28.90	*23.74*	55.4
22.55	27.85	*22.85*	28.21	*23.15*	28.58	*23.47*	28.97	*23.80*	55.5
22.60	27.92	*22.90*	28.28	*23.21*	28.65	*23.53*	29.04	*23,86*	55.6
22.66	27.98	*22.96*	28.34	*23.26*	28.72	*23.58*	29.11	*23.91*	55.7
22.71	28.05	*23.01*	28.41	*23.32*	28.78	*23.64*	29.18	*23.97*	55.8
22.76	28.11	*23.07*	28.48	*23.38*	28.85	*23.70*	29.24	*24.03*	55.9
22.82	28.18	*23.12*	28.54	*23.43*	28.92	*23.75*	29.31	*24.09*	56.0
22.87	28.24	*23.18*	28.61	*23.49*	28.99	*23.81*	29.38	*24.14*	56.1
22.92	28.31	*23.23*	28.68	*23.54*	29.05	*23.87*	29.45	*24.20*	56.2
22.98	28.37	*23.29*	28.74	*23.60*	29.12	*23.93*	29.52	*24.26*	56.3
23.03	28.44	*23.34*	28.81	*23.66*	29.19	*23.98*	29.58	*24.32*	56.4
23.09	28.50	*23.40*	28.87	*23.71*	29.26	*24.04*	29.65	*24.38*	56.5
23.14	28.56	*23.45*	28.94	*23.77*	29.32	*24.10*	29.72	*24.43*	56.6
23.20	28.63	*23.51*	29.01	*23.83*	29.39	*24.15*	29.79	*24.49*	56.7
23.25	28.69	*23.56*	29.07	*23.88*	29.46	*24.21*	29.86	*24.55*	56.8
23.31	28.76	*23.62*	29.14	*23.94*	29.53	*24.27*	29.93	*24.61*	56.9
23.36	28.82	*23.67*	29.20	*23.99*	29.59	*24.32*	29.99	*24.66*	57.0
23.42	28.89	*23.73*	29.27	*24.05*	29.66	*24.38*	30.06	*24.72*	57.1
23.47	28.95	*23.78*	29.34	*24.11*	29.73	*24.44*	30.13	*24.78*	57.2
23.52	29.02	*23.84*	29.40	*24.16*	29.80	*24.49*	30.20	*24.84*	57.3
23.58	29.08	*23.90*	29.47	*24.22*	29.86	*24.55*	30.27	*24.90*	57.4
23.63	29.15	*23.95*	29.53	*24.27*	29.93	*24.61*	30.34	*24.95*	57.5
23.69	29.21	*24.01*	29.60	*24.33*	30.00	*24.66*	30.41	*25.01*	57.6
23.74	29.28	*24.06*	29.66	*24.39*	30.07	*24.72*	30.48	*25.07*	57.7
23.80	29.34	*24.12*	29.73	*24.44*	30.14	*24.78*	30.55	*25.13*	57.8
23.85	29.41	*24.17*	29.80	*24.50*	30.20	*24.83*	30.62	*25.19*	57.9

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
58.0	27.87	*22.86*	28.03	*23.00*	28.38	*23.29*	28.73	*23.59*	29.10
58.1	27.93	*22.91*	28.09	*23.05*	28.44	*23.34*	28.79	*23.65*	29.17
58.2	27.99	*22.96*	28.15	*23.10*	28.50	*23.40*	28.86	*23.70*	29.23
58.3	28.05	*23.02*	28.21	*23.16*	28.56	*23.45*	28.92	*23.75*	29.29
58.4	28.11	*23.07*	28.28	*23.21*	28.62	*23.50*	28.98	*23.81*	29.36
58.5	28.17	*23.12*	28.34	*23.26*	28.69	*23.56*	29.04	*23.86*	29.42
58.6	28.23	*23.17*	28.40	*23.31*	28.75	*23.61*	29.11	*23.91*	29.48
58.7	28.29	*23.22*	28.46	*23.36*	28.81	*23.66*	29.17	*23.97*	29.55
58.8	28.35	*23.27*	28.52	*23.41*	28.88	*23.71*	29.23	*24.02*	29.61
58.9	28.41	*23.32*	28.58	*23.47*	28.94	*23.77*	29.30	*24.08*	29.68
59.0	28.47	*23.37*	28.64	*23.52*	29.00	*23.82*	29.36	*24.13*	29.74
59.1	28.53	*23.43*	28.71	*23.57*	29.06	*23.87*	29.42	*24.18*	29.80
59.2	28.59	*23.48*	28.77	*23.62*	29.12	*23.93*	29.49	*24.24*	29.87
59.3	28.65	*23.53*	28.83	*23.67*	29.19	*23.98*	29.55	*24.29*	29.93
59.4	28.71	*23.58*	28.89	*23.73*	29.25	*24.03*	29.61	*24.34*	29.99
59.5	28.78	*23.63*	28.95	*23.78*	29.31	*24.08*	29.68	*24.40*	30.06
59.6	28.84	*23.68*	29.01	*23.83*	29.37	*24.14*	29.74	*24.45*	30.13
59.7	28.90	*23.73*	29.07	*23.88*	29.43	*24.19*	29.80	*24.50*	30.19
59.8	28.96	*23.79*	29.13	*23.93*	29.50	*24.24*	29.87	*24.56*	30.26
59.9	29.02	*23.84*	29.20	*23.98*	29.56	*24.30*	29.93	*24.61*	30.32
60.0	29.08	*23.89*	29.26	*24.04*	29.62	*24.35*	29.99	*24.67*	30.39
60.1	29.14	*23.94*	29.32	*24.09*	29.68	*24.40*	30.06	*24.72*	30.45
60.2	29.20	*23.99*	29.38	*24.14*	29.74	*24.46*	30.12	*24.77*	30.52
60.3	29.26	*24.04*	29.44	*24.19*	29.81	*24.51*	30.19	*24.83*	30.59
60.4	29.32	*24.10*	29.50	*24.25*	29.87	*24.56*	30.25	*24.88*	30.65
60.5	29.38	*24.15*	29.56	*24.30*	29.93	*24.61*	30.32	*24.94*	30.72
60.6	29.45	*24.20*	29.63	*24.35*	29.99	*24.67*	30.38	*24.99*	30.78
60.7	29.51	*24.25*	29.69	*24.40*	30.06	*24.72*	30.45	*25.04*	30.85
60.8	29.57	*24.30*	29.75	*24.46*	30.12	*24.77*	30.51	*25.10*	30.91
60.9	29.63	*24.35*	29.81	*24.51*	30.18	*24.83*	30.57	*25.16*	30.98
61.0	29.69	*24.41*	29.87	*24.56*	30.25	*24.88*	30.64	*25.21*	31.05
61.1	29.75	*24.46*	29.93	*24.61*	30.31	*24.93*	30.70	*25.27*	31.11
61.2	29.81	*24.51*	29.99	*24.66*	30.38	*24.98*	30.77	*25.32*	31.18
61.3	29.87	*24.56*	30.06	*24.72*	30.44	*25.04*	30.83	*25.38*	31.25
61.4	29.93	*24.61*	30.12	*24.77*	30.50	*25.09*	30.90	*25.44*	31.32
61.5	29.99	*24.67*	30.18	*24.82*	30.57	*25.15*	30.96	*25.49*	31.39
61.6	30.06	*24.72*	30.25	*24.87*	30.63	*25.20*	31.03	*25.55*	31.45
61.7	30.12	*24.77*	30.31	*24.93*	30.69	*25.26*	31.09	*25.60*	31.52
61.8	30.18	*24.82*	30.37	*24.98*	30.76	*25.31*	31.16	*25.66*	31.59
61.9	30.25	*24.88*	30.44	*25.03*	30.82	*25.37*	31.23	*25.71*	31.66
62.0	30.31	*24.93*	30.50	*25,09*	30.89	*25.43*	31.29	*25.77*	31.72
62.1	30.37	*24.98*	30.56	*25.14*	30.95	*25.48*	31.36	*25.83*	31.79
62.2	30.43	*25.03*	30.63	*25.20*	31.01	*25.54*	31.43	*25.88*	31.86
62.3	30.50	*25.09*	30.69	*25.25*	31.08	*25.59*	31.49	*25.94*	31.93
62.4	30.56	*25.14*	30.75	*25.31*	31.14	*25.65*	31.56	*25.99*	31.99
62.5	30.62	*25.20*	30.82	*25.36*	31.21	*25.70*	31.63	*26.05*	32.06
62.6	30.69	*25.25*	30.88	*25.42*	31.28	*25.76*	31.69	*26.11*	32.13
62.7	30.75	*25.31*	30.94	*25.47*	31.34	*25.81*	31.76	*26.17*	32.20
62.8	30.81	*25.36*	31.01	*25.53*	31.41	*25.87*	31.83	*26.23*	32.27
62.9	30.87	*25.42*	31.07	*25.58*	31.47	*25.92*	31.89	*26.29*	32.34

Table.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		SCALE READING
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	
23.91	29.47	*24.23*	29.87	*24.55*	30.27	*24.89*	30.69	*25.25*	58.0
23.96	29.54	*24.28*	29.93	*24.61*	30.34	*24.95*	30.76	*25.31*	58.1
24.02	29.60	*24.34*	29.99	*24.66*	30.41	*25.01*	30.83	*25.37*	58.2
24.07	29.67	*24.39*	30.06	*24.72*	30.48	*25.07*	30.90	*25.43*	58.3
24.13	29.73	*24.45*	30.13	*24.78*	30.54	*25.13*	30.97	*25.49*	58.4
24.18	29.80	*24.50*	30.20	*24.83*	30.61	*25.18*	31.04	*25.55*	58.5
24.23	29.86	*24.56*	30.26	*24.89*	30.68	*25.23*	31.11	*25.61*	58.6
24.29	29.93	*24.61*	30.33	*24.94*	30.75	*25.29*	31.18	*25.67*	58.7
24.34	29.99	*24.67*	30.40	*25.00*	30.82	*25.35*	31.25	*25.73*	58.8
24.40	30.06	*24.72*	30.47	*25.06*	30.88	*25.41*	31.32	*25.80*	58.9
24.45	30.13	*24.78*	30.53	*25.12*	30.95	*25.47*	31.40	*25.86*	59.0
24.51	30.19	*24.83*	30.60	*25.18*	31.02	*25.53*	31.47	*25.92*	59.1
24.56	30.26	*24.89*	30.67	*25.24*	31.09	*25.59*	31.54	*25.98*	59.2
24.61	30.33	*24.95*	30.74	*25.30*	31.16	*25.65*	31.61	*26.04*	59.3
24.67	30.39	*25.00*	30.81	*25.36*	31.23	*25.71*	31.68	*26.10*	59.4
24.72	30.46	*25.06*	30.87	*25.41*	31.30	*25.77*	31.76	*26.16*	59.5
24.78	30.53	*25.11*	30.94	*25.47*	31.38	*25.83*	31.83	*26.23*	59.6
24.83	30.59	*25.17*	31.01	*25.53*	31.45	*25.89*	31.90	*26.29*	59.7
24.89	30.66	*25.23*	31.08	*25.59*	31.52	*25.95*	31.97	*26.35*	59.8
24.94	30.73	*25.29*	31.15	*25.65*	31.59	*26.02*	32.04	*26.42*	59.9
24.99	30.79	*25.34*	31.22	*25.71*	31.66	*26.08*	32.12	*26.48*	60.0
25.05	30.86	*25.40*	31.29	*25.77*	31.73	*26.14*	32.19	*26.54*	60.1
25.11	30.93	*25.46*	31.36	*25.83*	31.80	*26.20*	32.27	*26.61*	60.2
25.16	30.99	*25.52*	31.43	*25.89*	31.87	*26.27*	32.34	*26.67*	60.3
25.22	31.06	*25.57*	31.50	*25.95*	31.94	*26.33*	32.41	*26.73*	60.4
25.28	31.13	*25.63*	31.57	*26.01*	32.02	*26.39*	32.49	*26.80*	60.5
25.34	31.20	*25.69*	31.64	*26.07*	32.09	*26.45*	32.56	*26.86*	60.6
25.39	31.27	*25.75*	31.71	*26.13*	32.16	*26.52*	32.64	*26.92*	60.7
25.45	31.33	*25.80*	31.78	*26.19*	32.23	*26.58*	32.71	*26.99*	60.8
25.51	31.40	*25.86*	31.85	*26.25*	32.30	*26.64*	32.78	*27.05*	60.9
25.56	31.47	*25.92*	31.92	*26.31*	32.38	*26.70*	32.86	*27.12*	61.0
25.62	31.54	*25.98*	31.99	*26.37*	32.45	*26.76*	32.93	*27.18*	61.1
25.68	31.61	*26.04*	32.06	*26.43*	32.52	*26.83*	33.01	*27.24*	61.2
25.73	31.67	*26.10*	32.13	*26.49*	32.59	*26.89*	33.08	*27.31*	61.3
25.79	31.74	*26.16*	32.20	*26.55*	32.67	*26.95*	33.16	*27.37*	61.4
25.85	31.81	*26.22*	32.27	*26.61*	32.74	*27.01*	33.23	*27.44*	61.5
25.90	31.88	*26.28*	32.34	*26.67*	32.81	*27.08*	33.31	*27.50*	61.6
25.96	31.95	*26.34*	32.41	*27.73*	32.88	*27.14*	33.38	*27.56*	61.7
26.02	32.01	*26.40*	32.49	*26.79*	32.96	*27.20*	33.46	*27.63*	61.8
26.08	32.09	*26.46*	32.56	*26.85*	33.03	*27.27*	33.53	*27.69*	61.9
26.14	32.16	*26.51*	32.63	*26.92*	33.10	*27.33*	33.60	*27.76*	62.0
26.20	32.23	*26.57*	32.70	*26.98*	33.18	*27.39*	33.68	*27.82*	62.1
26.25	32.30	*26.63*	32.77	*27.04*	33.25	*27.46*	33.75	*27.88*	62.2
26.31	32.37	*26.69*	32.84	*27.10*	33.33	*27.52*	33.83	*27.95*	62.3
26.37	32.44	*26.75*	32.91	*27.16*	33.40	*27.58*	33.90	*28.01*	62.4
26.43	32.51	*26.81*	32.99	*27.23*	33.47	*27.65*	33.98	*28.08*	62.5
26.49	32.58	*26.87*	33.06	*27.29*	33.55	*27.71*	34.05	*28.15*	62.6
26.55	32.65	*26.93*	33.13	*27.35*	33.62	*27.77*	34.13	*28.22*	62.7
26.61	32.72	*26.99*	33.20	*27.41*	33.70	*27.84*	34.21	*28.28*	62.8
26.67	32.79	*27.06*	33.28	*27.48*	33.77	*27.90*	34.29	*28.35*	62.9

8 ALCOHOL

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
63.0	30.94	*25.47*	31.14	*25.64*	31.54	*25.98*	31.96	*26.35*	32.41
63.1	31.00	*25.52*	31.20	*25.69*	31.61	*26.03*	32.03	*26.41*	32.48
63.2	31.06	*25.58*	31.26	*25.75*	31.67	*26.09*	32.10	*26.46*	32.55
63.3	31.13	*25.63*	31.33	*25.80*	31.74	*26.15*	32.17	*26.52*	32.62
63.4	31.19	*25.69*	31.39	*25.86*	31.80	*26.21*	32.23	*26.58*	32.69
63.5	31.26	*25.74*	31.46	*25.91*	31.87	*26.26*	32.30	*26.64*	32.76
63.6	31.32	*25.80*	31.52	*25.97*	31.93	*26.32*	32.37	*26.70*	32.83
63.7	31.38	*25.85*	31.59	*26.02*	32.00	*26.38*	32.44	*26.76*	32.90
63.8	31.45	*25.91*	31.65	*26.08*	32.07	*26.44*	32.51	*26.82*	32.97
63.9	31.51	*25.96*	31.72	*26.13*	32.13	*26.49*	32.58	*26.88*	33.04
64.0	31.58	*26.02*	31.78	*26.19*	32.20	*26.55*	32.65	*26.94*	33.11
64.1	31.64	*26.07*	31.85	*26.25*	32.27	*26.61*	32.72	*26.99*	33.18
64.2	31.70	*26.13*	31.91	*26.30*	32.34	*26.67*	32.79	*27.05*	33.25
64.3	31.77	*26.18*	31.97	*26.36*	32.40	*26.72*	32.86	*27.11*	33.32
64.4	31.83	*26.24*	32.04	*26.41*	32.47	*26.78*	32.92	*27.17*	33.39
64.5	31.90	*26.29*	32.11	*26.47*	32.54	*26.84*	32.99	*27.23*	33.46
64.6	31.96	*26.35*	32.17	*26.53*	32.60	*26.90*	33.06	*27.29*	33.53
64.7	32.03	*26.40*	32.24	*26.58*	32.67	*26.95*	33.13	*27.35*	33.60
64.8	32.09	*26.46*	32.30	*26.64*	32.74	*27.01*	33.20	*27.41*	33.67
64.9	32.16	*26.51*	32.37	*26.69*	32.81	*27.07*	33.27	*27.47*	33.74
65.0	32.22	*26.57*	32.43	*26.75*	32.87	*27.13*	33.34	*27.53*	33.82
65.1	32.29	*26.63*	32.50	*26.80*	32.94	*27.19*	33.41	*27.59*	33.89
65.2	32.35	*26.68*	32.57	*26.86*	33.01	*27.25*	33.48	*27.65*	33.96
65.3	32.42	*26.74*	32.63	*26.92*	33.08	*27.31*	33.55	*27.71*	34.03
65.4	32.48	*26.79*	32.70	*26.97*	33.15	*27.37*	33.62	*27.77*	34.10
65.5	32.55	*26.85*	32.76	*27.03*	33.22	*27.43*	33.69	*27.83*	34.18
65.6	32.61	*26.90*	32.83	*27.09*	33.28	*27.49*	33.76	*27.89*	34.25
65.7	32.68	*29.96*	32.89	*27.15*	33.35	*27.54*	33.83	*27.95*	34.32
65.8	32.75	*27.01*	32.96	*27.21*	33.42	*27.60*	33.90	*28.01*	34.40
65.9	32.81	*27.07*	33.03	*27.26*	33.49	*27.66*	33.97	*28.07*	34.47
66.0	32.88	*27.13*	33.10	*27.32*	33.56	*27.72*	34.04	*28.13*	34.54
66.1	32.94	*27.19*	33.17	*27.38*	33.63	*27.78*	34.11	*28.19*	34.62
66.2	33.01	*27.25*	33.23	*27.44*	33.70	*27.84*	34.18	*28.26*	34.69
66.3	33.07	*27.30*	33.30	*27.50*	33.77	*27.90*	34.25	*28.32*	34.76
66.4	33.14	*27.36*	33.37	*27.56*	33.84	*27.96*	34.33	*28.38*	34.84
66.5	33.21	*27.42*	33.44	*27.62*	33.91	*28.02*	34.40	*28.45*	34.91
66.6	33.28	*27.48*	33.51	*27.68*	33.98	*28.08*	34.47	*28.51*	34.99
66.7	33.35	*27.54*	33.58	*27.73*	34.05	*28.14*	34.54	*28.57*	35.06
66.8	33.41	*27.60*	33.65	*27.79*	34.12	*28.20*	34.62	*28.64*	35.14
66.9	33.48	*27.65*	33.72	*27.85*	34.19	*28.27*	34.69	*28.70*	35.21
67.0	33.55	*27.71*	33.79	*27.91*	34.26	*28.33*	34.76	*28.76*	35.29
67.1	33.62	*27.77*	33.86	*27.97*	34.34	*28.39*	34.83	*28.82*	35.37
67.2	33.69	*27.83*	33.92	*28.03*	34.41	*28.45*	34.91	*28.89*	35.44
67.3	33.76	*27.89*	33.99	*28.09*	34.48	*28.52*	34.98	*28.95*	35.52
67.4	33.82	*27.95*	34.06	*28.15*	34.55	*28.58*	35.05	*29.01*	35.60
67.5	33.89	*28.01*	34.13	*28.21*	34.62	*28.64*	35.13	*29.08*	35.67
67.6	33.96	*28.06*	34.20	*28.27*	34.69	*28.70*	35.20	*29.14*	35.75
67.7	34.03	*28.12*	34.27	*28.34*	34.76	*28.76*	35.28	*29.21*	35.82
67.8	34.09	*28.18*	34.34	*28.40*	34.84	*28.83*	35.35	*29.27*	35.90
67.9	34.16	*28.24*	34.41	*28.46*	34.91	*28.89*	35.43	*29.34*	35.98

TABLE.—Continued.

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
26.73	32.87	*27.12*	33.35	*27.54*	33.84	*27.96*	34.36	*28.42*	63.0
26.79	32.94	*27.18*	33.42	*27.60*	33.92	*28.03*	34.44	*28.49*	63.1
26.85	33.01	*27.24*	33.50	*27.66*	33.99	*28.09*	34.52	*28.55*	63.2
26.91	33.08	*27.31*	33.57	*27.72*	34.07	*28.16*	34.60	*28.62*	63.3
26.96	33.15	*27.37*	33.64	*27.79*	34.15	*28.23*	34.67	*28.69*	63.4
27.02	33.22	*27.43*	33.72	*27.85*	34.22	*28.29*	34.75	*28.76*	63.5
27.09	33.30	*27.49*	33.79	*27.91*	34.30	*28.36*	34.83	*28.82*	63.6
27.15	33.37	*27.56*	33.86	*27.98*	34.38	*28.43*	34.91	*28.89*	63.7
27.21	33.44	*27.62*	33.93	*28.04*	34.45	*28.49*	34.98	*28.96*	63.8
27.27	33.51	*27.68*	34.01	*28.11*	34.53	*28.56*	35.07	*29.03*	63.9
27.33	33.59	*27.74*	34.08	*28.17*	34.61	*28.63*	35.15	*29.10*	64.0
27.39	33.66	*27.81*	34.16	*28.24*	34.68	*28.69*	35.23	*29.17*	64.1
27.45	33.73	*27.87*	34.23	*28.30*	34.76	*28.76*	35.31	*29.24*	64.2
27.51	33.80	*27.93*	34.31	*28.37*	34.84	*28.83*	35.39	*29.31*	64.3
27.57	33.88	*27.99*	34.39	*28.43*	34.92	*28.89*	35.48	*29.38*	64.4
27.63	33.95	*28.06*	34.46	*28.50*	34.99	*28.96*	35.56	*29.45*	64.5
27.70	34.02	*28.12*	34.54	*28.57*	35.07	*29.03*	35.64	*29.52*	64.6
27.76	34.10	*28.19*	34.61	*28.63*	35.15	*29.10*	35.72	*29.60*	64.7
27.82	34.17	*28.25*	34.69	*28.70*	35.23	*29.17*	35.80	*29.67*	64.8
27.88	34.24	*28.31*	34.76	*28.76*	35.31	*29.24*	35.89	*29.74*	64.9
27.94	34.32	*28.38*	34.84	*28.83*	35.39	*29.31*	35.97	*29.81*	65.0
28.00	34.39	*28.44*	34.92	*28.89*	35.47	*29.38*	36.05	*29.88*	65.1
28.06	34.47	*28.51*	34.99	*28.96*	35.55	*29.44*	36.13	*29.95*	65.2
28.13	34.54	*28.57*	35.07	*29.03*	35.63	*29.51*	36.21	*30.02*	65.3
28.19	34.61	*28.63*	35.15	*29.10*	35.71	*29.58*	36.30	*30.10*	65.4
28.25	34.69	*28.70*	35.23	*29.16*	35.79	*29.65*	36.38	*30.17*	65.5
28.32	34.76	*28.76*	35.30	*29.23*	35.87	*29.72*	36.46	*30.24*	65.6
28.38	34.84	*28.83*	35.38	*29.30*	35.94	*29.79*	36.55	*30.32*	65.7
28.45	34.91	*28.89*	35.46	*29.37*	36.02	*29.86*	36.63	*30.39*	65.8
28.51	34.99	*28.96*	35.54	*29.44*	36.11	*29.93*	36.71	*30.46*	65.9
28.57	35.06	*29.02*	35.62	*29.51*	36.19	*30.00*	36.79	*30.54*	66.0
28.64	35.14	*29.09*	35.70	*29.58*	36.27	*30.07*	36.88	*30.61*	66.1
28.70	35.22	*29.16*	35.77	*29.64*	36.35	*30.15*	36.96	*30.68*	66.2
28.76	35.30	*29.23*	35.85	*29.71*	36.43	*30.22*	37.04	*30.76*	66.3
28.83	35.38	*29.29*	35.93	*29.78*	36.52	*30.29*	37.13	*30.83*	66.4
28.89	35.45	*29.36*	36.01	*29.85*	36.60	*30.36*	37.22	*30.90*	66.5
28.96	35.53	*29.43*	36.09	*29.92*	36.68	*30.43*	37.30	*30.98*	66.6
29.03	35.61	*29.50*	36.17	*29.99*	36.76	*30.51*	37.39	*31.05*	66.7
29.09	35.69	*29.57*	36.25	*30.06*	36.84	*30.58*	37.48	*31.13*	66.8
29.15	35.77	*29.64*	36.33	*30.13*	36.93	*30.65*	37.57	*31.21*	66.9
29.22	35.84	*29.71*	36.41	*30.20*	37.01	*30.72*	37.65	*31.28*	67.0
29.29	35.92	*29.77*	36.49	*30.27*	37.09	*30.80*	37.74	*31.36*	67.1
29.35	36.00	*29.84*	36.57	*30.34*	37.18	*30.87*	37.83	*31.44*	67.2
29.42	36.08	*29.91*	36.65	*30.41*	37.26	*30.94*	37.91	*31.51*	67.3
29.49	36.16	*29.98*	36.73	*30.49*	37.35	*31.02*	38.00	*31.59*	67.4
29.55	36.24	*30.05*	36.81	*30.56*	37.44	*31.09*	38.09	*31.65*	67.5
29.62	36.32	*30.12*	36.90	*30.63*	37.52	*31.17*	38.18	*31.74*	67.6
29.69	36.40	*30.19*	36.98	*30.70*	37.61	*31.24*	38.26	*31.82*	67.7
29.75	36.48	*30.26*	37.06	*30.77*	37.69	*31.32*	38.35	*31.89*	67.8
29.82	36.56	*30.33*	37.14	*30.84*	37.78	*31.39*	38.44	*31.97*	67.9

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
68.0	34.23	*28.30*	34.48	*28.52*	34.98	*28.95*	35.50	*29.41*	36.05
68.1	34.30	*28.36*	34.55	*28.58*	35.05	*29.01*	35.57	*29.47*	36.13
68.2	34.36	*28.42*	34.62	*28.64*	35.13	*29.08*	35.65	*29.54*	36.21
68.3	34.43	*28.48*	34.69	*28.70*	35.20	*29.14*	35.72	*29.60*	36.29
68.4	34.50	*28.54*	34.76	*28.76*	35.27	*29.21*	35.80	*29.67*	36.37
68.5	34.57	*28.59*	34.83	*28.82*	35.35	*29.27*	35.87	*29.73*	36.45
68.6	34.64	*28.65*	34.90	*28.88*	35.42	*29.33*	35.95	*29.80*	36.52
68.7	34.70	*28.71*	34.97	*28.95*	35.49	*29.40*	36.02	*29.86*	36.60
68.8	34.77	*28.77*	35.04	*29.01*	35.57	*29.46*	36.10	*29.93*	36.68
68.9	34.84	*28.83*	35.12	*29.07*	35.64	*29.53*	36.18	*29.99*	36.76
69.0	34.91	*28.89*	35.19	*29.13*	35.71	*29.59*	36.25	*30.06*	36.84
69.1	34.97	*28.95*	35.26	*29.19*	35.79	*29.65*	36.33	*30.13*	36.91
69.2	35.04	*29.01*	35.33	*29.26*	35.86	*29.72*	36.41	*30.20*	36.99
69.3	35.12	*29.07*	35.40	*29.32*	35.93	*29.78*	36.48	*30.27*	37.07
69.4	35.19	*29.14*	35.47	*29.38*	36.01	*29.85*	36.56	*30.33*	37.15
69.5	35.27	*29.20*	35.55	*29.45*	36.08	*29.91*	36.64	*30.40*	37.24
69.6	35.34	*29.26*	35.62	*29.51*	36.16	*29.97*	36.72	*30.47*	37.32
69.7	35.41	*29.33*	35.69	*29.57*	36.23	*30.04*	36.79	*30.54*	37.40
69.8	35.49	*29.39*	35.76	*29.64*	36.31	*30.11*	36.87	*30.61*	37.48
69.9	35.56	*29.46*	35.83	*29.70*	36.39	*30.17*	36.95	*30.67*	37.56
70.0	35.64	*29.52*	35.91	*29.76*	36.46	*30.24*	37.02	*30.74*	37.64
70.1	35.71	*29.59*	35.98	*29.82*	36.54	*30.31*	37.10	*30.81*	37.72
70.2	35.78	*29.65*	36.05	*29.89*	36.61	*30.38*	37.19	*30.88*	37.80
70.3	35.86	*29.72*	36.13	*29.95*	36.69	*30.44*	37.27	*30.95*	37.89
70.4	35.93	*29.78*	36.20	*30.01*	36.76	*30.51*	37.35	*31.01*	37.97
70.5	36.01	*29.85*	36.28	*30.08*	36.84	*30.58*	37.43	*31.09*	38.05
70.6	36.08	*29.91*	36.35	*30.15*	36.92	*30.64*	37.51	*31.16*	38.13
70.7	36.16	*29.97*	36.43	*30.21*	36.99	*30.71*	37.59	*31.23*	38.22
70.8	36.23	*30.04*	36.50	*30.28*	37.07	*30.78*	37.67	*31.30*	38.30
70.9	36.31	*30.11*	36.58	*30.35*	37.15	*30.85*	37.75	*31.37*	38.38
71.0	36.38	*30.17*	36.65	*30.41*	37.23	*30.91*	37.83	*31.44*	38.47
71.1	36.46	*30.24*	36.73	*30.48*	37.31	*30.98*	37.91	*31.51*	38.55
71.2	36.53	*30.30*	36.80	*30.55*	37.39	*31.05*	37.99	*31.59*	38.63
71.3	36.60	*30.37*	36.88	*30.61*	37.47	*31.12*	38.07	*31.66*	38.72
71.4	36.68	*30.44*	36.95	*30.68*	37.55	*31.19*	38.16	*31.73*	38.80
71.5	36.75	*30.50*	37.03	*30.75*	37.63	*31.26*	38.24	*31.80*	38.88
71.6	36.83	*30.57*	37.11	*30.81*	37.71	*31.33*	38.32	*31.87*	38.97
71.7	36.90	*30.64*	37.19	*30.88*	37.79	*31.40*	38.40	*31.94*	39.05
71.8	36.98	*30.70*	37.27	*30.95*	37.87	*31.47*	38.49	*32.01*	39.14
71.9	37.05	*30.77*	37.34	*31.01*	37.94	*31.54*	38.57	*32.09*	39.23
72.0	37.13	*30.84*	37.42	*31.08*	38.02	*31.61*	38.65	*32.17*	39.31
72.1	37.21	*30.90*	37.50	*31.15*	38.11	*31.68*	38.74	*32.24*	39.40
72.2	37.29	*30.97*	37.58	*31.22*	38.19	*31.75*	38.82	*32.32*	39.49
72.3	37.36	*31.03*	37.66	*31.29*	38.27	*31.82*	38.90	*32.39*	39.57
72.4	37.44	*31.10*	37.73	*31.36*	38.35	*31.89*	38.98	*32.47*	39.66
72.5	37.52	*31.17*	37.81	*31.42*	38.43	*31.96*	39.07	*32.54*	39.75
72.6	37.60	*31.24*	37.89	*31.49*	38.51	*32.04*	39.16	*32.62*	39.83
72.7	37.67	*31.31*	37.97	*31.56*	38.59	*32.11*	39.24	*32.69*	39.92
72.8	37.75	*31.37*	38.05	*31.63*	38.67	*32.18*	39.33	*32.77*	40.01
72.9	37.83	*31.44*	38.13	*31.70*	38.76	*32.26*	39.41	*32.84*	40.10

Table.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
29.89	36.63	30.40	37.23	30.91	37.86	31.47	38.53	32.05	68.0
29.95	36.71	30.47	37.31	30.99	37.95	31.54	38.61	32.13	68.1
30.02	36.79	30.54	37.39	31.06	38.03	31.62	38.70	32.21	68.2
30.09	36.87	30.61	37.48	31.13	38.12	31.69	38.79	32.29	68.3
30.16	36.95	30.68	37.56	31.21	38.21	31.77	38.88	32.37	68.4
30.23	37.03	30.75	37.65	31.28	38.30	31.84	38.96	32.45	68.5
30.30	37.12	30.82	37.73	31.35	38.38	31.92	39.06	32.53	68.6
30.37	37.20	30.89	37.82	31.43	38.47	31.99	39.15	32.61	68.7
30.43	37.28	30.96	37.90	31.50	38.56	32.07	39.24	32.69	68.8
30.50	37.36	31.03	37.98	31.57	38.64	32.15	39.33	32.77	68.9
30.57	37.45	31.10	38.07	31.65	38.73	32.23	39.43	32.86	69.0
30.64	37.53	31.17	38.15	31.72	38.82	32.31	39.52	32.94	69.1
30.71	37.61	31.25	38.24	31.79	38.90	32.39	39.61	33.02	69.2
30.78	37.69	31.32	38.32	31.87	38.99	32.47	39.70	33.10	69.3
30.85	37.78	31.39	38.41	31.94	39.08	32.55	39.80	33.18	69.4
30.92	37.86	31.46	38.50	32.02	39.17	32.63	39.89	33.26	69.5
30.99	37.94	31.54	38.58	32.09	39.26	32.71	39.98	33.34	69.6
31,06	38.03	31.61	38.67	32.17	39.35	32.78	40.07	33.43	69.7
31.13	38.11	31.68	38.75	32.25	39.45	32.86	40.17	33.51	69.8
31.20	38.19	31.75	38.84	32.33	39.54	32.95	40.26	33.59	69.9
31.27	38.28	31.83	38.92	32.41	39.63	33.02	40.35	33.67	70.0
31.35	38.36	31.90	39.01	32.49	39.72	33.11	40.44	33.75	70.1
31.42	38.45	31.97	39.10	32.57	39.81	33.19	40.53	33.84	70.2
31.49	38.53	32.05	39.19	32.65	39.90	33.27	40.62	33.92	70.3
31.56	38.61	32.12	39.28	32.72	39.99	33.35	40.72	34.00	70.4
31.63	38.70	32.20	39.37	32.80	40.08	33.43	40.81	34.08	70.5
31.70	38.78	32.28	39.46	32.88	40.17	33.51	40.90	34.17	70.6
31.78	38.87	32.36	39.55	32.96	40.26	33.59	40.99	34.25	70.7
31.85	38.95	32.43	39.64	33.04	40.35	33.68	41.08	34.33	70.8
31.92	39.04	32.51	39.73	33.12	40.45	33.76	41.18	34.42	70.9
31.99	39.12	32.59	39.82	33.20	40.54	33.84	41.27	34.50	71.0
32.07	39.21	32.67	39.91	33.28	40.63	33.92	41.36	34.58	71.1
32.15	39.30	32.74	40.00	33.36	40.72	34.00	41.46	34.67	71.2
32.22	39.39	32.82	40.09	33.44	40.81	34.08	41.55	34.75	71.3
32.30	39.48	32.90	40.18	33.52	40.90	34.17	41.64	34.83	71.4
32.37	39.57	32.98	40.27	33.60	40.99	34.25	41.74	34.92	71.5
32.45	39.65	33.05	40.36	33.68	41.08	34.33	41.83	35.00	71.6
32.53	39.74	33.13	40.45	33.76	41.18	34.41	41.93	35.08	71.7
32.60	39.83	33.21	40.54	33.84	41.27	34.50	42.02	35.17	71.8
32.68	39.92	33.29	40.63	33.92	41.36	34.58	42.11	35.25	71.9
32.76	40.01	33.37	40.72	34.00	41.45	34.66	42.21	35.34	72.0
32.83	40.10	33.45	40.81	34.08	41.55	34.74	42.30	35.42	72.1
32.91	40.18	33.52	40.90	34.16	41.64	34.83	42.40	35.51	72.2
32.98	40.27	33.60	40.99	34.24	41.73	34.91	42.49	35.59	72.3
33.06	40.36	33.68	41.08	34.33	41.82	34.99	42.58	35.68	72.4
33.14	40.45	33.76	41.17	34.41	41.92	35.08	42.68	35.76	72.5
33.22	40.54	33.84	41.26	34.49	42.01	35.16	42.77	35.85	72.6
33.29	40.62	33.91	41.35	34.57	42.10	35.24	42.87	35.93	72.7
33.37	40.71	33.99	41.45	34.65	42.19	35.33	42.96	36.02	72.8
33.45	40.80	34.07	41.54	34.73	42.29	35.41	43.06	36.10	72.9

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
73.0	37.91	*31.54*	38.21	*31.77*	38.84	*32.33*	39.50	*32.92*	40.18
73.1	37.98	*31.58*	38.29	*31.84*	38.92	*32.40*	39.59	*32.99*	40.27
73.2	38.06	*31.65*	38.37	*31.90*	39.00	*32.48*	39.67	*33.07*	40.36
73.3	38.14	*31.71*	38.45	*31.97*	39.08	*32.55*	39.76	*33.15*	40.44
73.4	38.22	*31.78*	38.53	*32.04*	39.17	*32.62*	39.84	*33.22*	40.53
73.5	38.30	*31.85*	38.61	*32.12*	39.25	*32.70*	39.93	*33.30*	40.62
73.6	38.38	*31.92*	38.69	*32.19*	39.34	*32.77*	40.02	*33.37*	40.70
73.7	38.46	*31.99*	38.77	*32.26*	39.42	*32.85*	40.10	*33.45*	40.79
73.8	38.54	*32.06*	38.85	*32.34*	39.50	*32.92*	40.19	*33.53*	40.88
73.9	38.62	*32.13*	38.93	*32.41*	39.59	*32.99*	40.28	*33.60*	40.97
74.0	38.70	*32.20*	39.01	*32.48*	39.67	*33.07*	40.36	*33.68*	41.05
74.1	38.78	*32.27*	39.09	*32.55*	39.76	*33.15*	40.45	*33.76*	41.14
74.2	38.86	*32.35*	39.18	*32.63*	39.84	*33.22*	40.53	*33.83*	41.23
74.3	38.94	*32.42*	39.26	*32.70*	39.92	*33.30*	40.62	*33.91*	41.32
74.4	39.02	*32.49*	39.34	*32.77*	40.01	*33.37*	40.71	*33.98*	41.41
74.5	39.10	*32.56*	39.43	*32.85*	40.09	*33.45*	40.79	*34.06*	41.50
74.6	39.18	*32.63*	39.51	*32.92*	40.18	*33.53*	40.88	*34.14*	41.59
74.7	39.26	*32.70*	39.59	*32.99*	40.27	*33.60*	40.97	*34.22*	41.68
74.8	39.35	*32.78*	39.68	*33.07*	40.35	*33.68*	41.05	*34.30*	41.77
74.9	39.43	*32.85*	39.76	*33.15*	40.44	*33.76*	41.14	*34.38*	41.86
75.0	39.51	*32.92*	39.84	*33.22*	40.53	*33.83*	41.23	*34.46*	41.95
75.1	39.60	*32.99*	39.93	*33.30*	40.61	*33.91*	41.32	*34.54*	42.04
75.2	39.68	*33.07*	40.01	*33.37*	40.70	*33.98*	41.41	*34.61*	42.13
75.3	39.76	*33.15*	40.09	*33.45*	40.78	*34.06*	41.50	*34.69*	42.22
75.4	39.84	*33.22*	40.18	*33.53*	40.87	*34.14*	41.58	*34.77*	42.31
75.5	39.93	*33.30*	40.27	*33.60*	40.96	*34.22*	41.67	*34.85*	42.40
75.6	40.01	*33.37*	40.35	*33.68*	41.04	*34.30*	41.76	*34.93*	42.49
75.7	40.09	*33.45*	40.44	*33.76*	41.13	*34.38*	41.85	*35.01*	42.58
75.8	40.18	*33.53*	40.53	*33.83*	41.22	*34.45*	41.94	*35.09*	42.67
75.9	40.27	*33.60*	40.61	*33.91*	41.31	*34.53*	42.03	*35.17*	42.76
76.0	40.35	*33.68*	40.70	*33.98*	41.40	*34.61*	42.12	*35.25*	42.85
76.1	40.44	*33.76*	40.78	*34.06*	41.48	*34.68*	42.21	*35.33*	42.95
76.2	40.53	*33.83*	40.87	*34.14*	41.57	*34.77*	42.30	*35.41*	43.04
76.3	40.61	*33.91*	40.96	*34.22*	41.66	*34.84*	42.39	*35.50*	43.13
76.4	40.70	*33.98*	41.04	*34.29*	41.75	*34.92*	42.48	*35.58*	43.22
76.5	40.78	*34.06*	41.13	*34.37*	41.83	*35.00*	42.57	*35.66*	43.32
76.6	40.87	*34.14*	41.22	*34.45*	41.92	*35.08*	42.66	*35.74*	43.41
76.7	40.96	*34.22*	41.30	*34.53*	42.01	*35.16*	42.75	*35.82*	43.50
76.8	41.04	*34.29*	41.39	*34.60*	42.10	*35.24*	42.84	*35.90*	43.60
76.9	41.13	*34.37*	41.48	*34.68*	42.19	*35.32*	42.93	*35.98*	43.69
77.0	41.22	*34.45*	41.57	*34.76*	42.28	*35.40*	43.02	*36.07*	43.79
77.1	41.31	*34.52*	41.65	*34.84*	42.37	*35.48*	43.11	*36.15*	43.88
77.2	41.39	*34.60*	41.74	*34.91*	42.46	*35.56*	43.20	*36.24*	43.97
77.3	41.48	*34.68*	41.83	*34.99*	42.54	*35.64*	43.30	*36.32*	44.07
77.4	41.57	*34.75*	41.91	*35.07*	42.63	*35.72*	43.39	*36.40*	44.16
77.5	41.66	*34.83*	42.00	*35.15*	42.72	*35.80*	43.48	*36.49*	44.26
77.6	41.75	*34.91*	42.09	*35.23*	42.81	*35.88*	43.57	*36.57*	44.35
77.7	41.83	*34.98*	42.17	*35.30*	42.90	*35.96*	43.67	*36.66*	44.45
77.8	41.92	*35.06*	42.26	*35.38*	42.99	*36.04*	43.76	*36.74*	44.54
77.9	42.01	*35.14*	42.35	*35.46*	43.08	*36.13*	43.85	*36.82*	44.64

TABLE.—Continued. 8

21° C.	22° C.		23° C.		24° C.		25° C.		
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	SCALE READING
33.52	40.88	34.15	41.63	34.81	42.38	35.49	43.15	36.18	73.0
33.60	40.97	34.23	41.72	34.89	42.47	35.58	43.24	36.27	73.1
33.68	41.06	34.31	41.81	34.98	42.56	35.66	43.33	36.35	73.2
33.75	41.15	34.39	41.90	35.06	42.66	35.74	43.43	36.43	73.3
33.83	41.24	34.47	41.99	35.14	42.75	35.83	43.52	36.52	73.4
33.91	41.33	34.55	42.08	35.22	42.84	35.91	43.61	36.60	73.5
33.98	41.42	34.63	42.17	35.31	42.93	35.99	43.70	36.68	73.6
34.06	41.51	34.71	42.27	35.39	43.03	36.08	43.80	36.77	73.7
34.14	41.60	34.79	42.36	35.47	43.12	36.16	43.89	36.85	73.8
34.22	41.69	34.87	42.45	35.55	43.21	36.24	43.98	36.93	73.9
34.30	41.78	34.95	42.54	35.64	43.31	36.33	44.08	37.02	74.0
34.38	41.87	35.03	42.63	35.72	43.40	36.41	44.18	37.11	74.1
34.46	41.96	35.12	42.72	35.80	43.49	36.49	44.28	37.20	74.2
34.54	42.06	35.20	42.82	35.88	43.58	36.58	44.38	37.29	74.3
34.62	42.15	35.28	42.91	35.97	43.68	36.66	44.48	37.38	74.4
34.70	42.24	35.36	43.00	36.05	43.77	36.74	44.57	37.47	74.5
34.78	42.33	35.45	43.09	36.13	43.86	36.83	44.67	37.56	74.6
34.86	42.42	35.53	43.19	36.22	43.95	36.91	44.77	37.65	74.7
34.94	42.51	35.61	43.28	36.30	44.05	36.99	44.87	37.75	74.8
35.02	42.61	35.69	43.37	36.39	44.15	37.08	44.97	37.84	74.9
35.10	42.70	35.78	43.46	36.47	44.25	37.17	45.07	37.93	75.0
35.18	42.79	35.86	43.56	36.55	44.34	37.26	45.18	38.02	75.1
35.26	42.88	35.95	43.65	36.64	44.44	37.35	45.29	38.12	75.2
35.34	42.97	36.03	43.74	36.72	44.53	37.44	45.39	38.21	75.3
35.43	43.07	36.11	43.83	36.81	44.63	37.53	45.50	38.31	75.4
35.51	43.16	36.20	43.92	36.89	44.73	37.62	45.61	38.40	75.5
35.59	43.25	36.28	44.02	36.97	44.83	37.71	45.71	38.50	75.6
35.67	43.35	36.36	44.12	37.06	44.93	37.80	45.82	38.60	75.7
35.75	43.44	36.45	44.21	37.15	45.03	37.89	45.92	38.69	75.8
35.84	43.53	36.53	44.31	37.24	45.13	37.98	46.02	38.79	75.9
35.92	43.63	36.62	44.41	37.33	45.24	38.08	46.12	38.88	76.0
36.00	43.72	36.70	44.50	37.42	45.34	38.17	46.23	38.98	76.1
36.08	43.81	36.79	44.60	37.50	45.44	38.27	46.34	39.08	76.2
36.17	43.91	36.87	44.70	37.59	45.55	38.36	46.45	39.18	76.3
36.25	44.00	36.96	44.80	37.68	45.65	38.46	46.56	39.29	76.4
36.34	44.10	37.04	44.89	37.77	45.75	38.55	46.67	39.39	76.5
36.42	44.19	37.13	44.99	37.86	45.86	38.65	46.78	39.49	76.6
36.51	44.29	37.22	45.09	37.95	45.96	38.74	46.89	39.59	76.7
36.59	44.38	37.30	45.19	38.04	46.07	38.84	47.00	39.69	76.8
36.68	44.48	37.39	45.30	38.13	46.18	38.93	47.11	39.80	76.9
36.76	44.57	37.47	45.40	38.23	46.29	39.03	47.23	39.90	77.0
36.85	44.67	37.56	45.50	38.32	46.40	39.13	47.34	40.00	77.1
36.93	44.76	37.65	45.60	38.42	46.51	39.23	47.45	40.11	77.2
37.02	44.86	37.73	45.70	38.51	46.62	39.34	47.57	40.22	77.3
37.10	44.95	37.82	45.81	38.60	46.73	39.44	47.68	40.32	77.4
37.19	45.05	37.91	45.91	38.70	46.84	39.54	47.80	40.43	77.5
37.28	45.15	37.99	46.01	38.79	46.95	39.64	47.91	40.54	77.6
37.36	45.25	38.08	46.12	38.89	47.06	39.74	48.02	40.65	77.7
37.45	45.35	38.18	46.23	38.98	47.17	39.85	48.14	40.75	77.8
37.53	45.45	38.27	46.34	39.08	47.28	39.95	48.26	40.86	77.9

8 ALCOHOL

SCALE READING	17.5° C.		18° C.		19° C.		20° C.		21° C.
	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume
78.0	42.09	*35.22*	42.43	*35.54*	43.17	*36.21*	43.94	*36.91*	44.73
78.1	42.18	*35.30*	42.52	*35.62*	43.27	*36.29*	44.04	*36.99*	44.83
78.2	42.26	*35.38*	42.61	*35.70*	43.36	*36.38*	44.13	*37.08*	44.92
78.3	42.35	*35.45*	42.70	*35.77*	43.45	*36.46*	44.23	*37.16*	45.02
78.4	42.44	*35.53*	42.78	*35.85*	43.54	*36.54*	44.32	*37.25*	45.12
78.5	42.52	*35.61*	42.87	*35.93*	43.63	*36.63*	44.42	*37.33*	45.22
78.6	42.61	*35.69*	42.96	*36.01*	43.72	*36.71*	44.51	*37.42*	45.32
78.7	42.69	*35.77*	43.05	*36.09*	43.82	*36.79*	44.60	*37.50*	45.42
78.8	42.78	*35.84*	43.14	*36.17*	43.91	*36.88*	44.70	*37.59*	45.52
78.9	42.86	*35.92*	43.23	*36.26*	44.00	*36.96*	44.79	*37.68*	45.62
79.0	42.95	*36.00*	43.32	*36.34*	44.09	*37.04*	44.89	*37.76*	45.72
79.1	43.04	*36.08*	43.41	*36.42*	44.19	*37.13*	44.98	*37.85*	45.82
79.2	43.13	*36.16*	43.50	*36.50*	44.28	*37.21*	45.08	*37.94*	45.92
79.3	43.22	*36.25*	43.59	*36.59*	44.38	*37.30*	45.18	*38.02*	46.02
79.4	43.31	*36.33*	43.68	*36.67*	44.47	*37.38*	45.28	*38.11*	46.13
79.5	43.40	*36.41*	43.77	*36.75*	44.56	*37.47*	45.38	*38.20*	46.24
79.6	43.49	*36.49*	43.86	*36.83*	44.65	*37.56*	45.48	*38.30*	46.34
79.7	43.58	*36.57*	43.95	*36.92*	44.75	*37.64*	45.58	*38.39*	46.45
79.8	43.67	*36.66*	44.05	*37.00*	44.84	*37.73*	45.68	*38.48*	46.56
79.9	43.76	*36.74*	44.14	*37.09*	44.93	*37.81*	45.78	*38.57*	46.67
80.0	43.85	*36.82*	44.24	*36.17*	45.04	*37.90*	45.88	*38.67*	46.77

TABLE.—Concluded.

8

21° C.	22° C.		23° C.		24° C.		25° C.		SCALE READING
Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	Per cent by volume	Per cent by weight	
37.62	45.56	*38.37*	46.45	*39.18*	47.40	*40.05*	48.37	*40.97*	78.0
37.71	45.66	*38.46*	46.56	*39.29*	47.51	*40.16*	48.49	*41.08*	78.1
37.79	45.76	*38.56*	46.67	*39.39*	47.63	*40.27*	48.60	*41.18*	78.2
37.88	45.86	*38.65*	46.78	*39.49*	47.74	*40.37*	48.72	*41.29*	78.3
37.97	45.96	*38.75*	46.89	*39.59*	47.85	*40.48*	48.84	*41.40*	78.4
38.06	46.07	*38.84*	47.00	*39.69*	47.97	*40.59*	48.95	*41.51*	78.5
38.15	46.17	*38.93*	47.11	*39.80*	48.08	*40.69*	49.07	*41.62*	78.6
38.24	46.28	*39.03*	47.22	*39.90*	48.19	*40.80*	49.19	*41.73*	78.7
38.33	46.39	*39.13*	47.34	*40.00*	48.31	*40.90*	49.31	*41.84*	78.8
38.43	46.50	*39.23*	47.45	*40.11*	48.42	*41.01*	49.42	*41.95*	78.9
38.52	46.61	*39.33*	47.56	*40.21*	48.53	*41.12*	49.54	*42.05*	79.0
38.61	46.72	*39.43*	47.67	*40.32*	48.65	*41.22*	49.66	*42.16*	79.1
38.70	46.83	*39.54*	47.79	*40.42*	48.76	*41.33*	49.77	*42.27*	79.2
38.80	46.93	*39.64*	47.90	*40.53*	48.88	*41.44*	49.89	*42.38*	79.3
38.89	47.04	*39.74*	48.01	*40.63*	48.99	*41.54*	50.01	*42.49*	79.4
38.98	47.15	*39.84*	48.12	*40.74*	49.10	*41.65*	50.13	*42.60*	79.5
39.08	47.26	*39.94*	48.23	*40.84*	49.22	*41.76*	50.24	*42.71*	79.6
39.18	47.37	*40.04*	48.34	*40.95*	49.33	*41.86*	50.36	*42.82*	79.7
39.28	47.48	*40.14*	48.46	*41.05*	49.45	*41.97*	50.48	*42.93*	79.8
39.38	47.59	*40.24*	48.57	*41.16*	49.56	*42.08*	50.59	*43.04*	79.9
39.48	47.70	*40.35*	48.68	*41.26*	49.68	*42.18*	50.71	*43.15*	80.0

9 *Temperature corrections to readings of Saccharometers (Standard at 20°C.).*

(This table is calculated using the data on thermal expansion of sugar solutions by Plato, assuming the instrument to be of Jena 16^{III} glass. The table should be used with caution and only for approximate results when the temperature differs much from the standard temperature or from the temperature of the surrounding air.)

TEMPERATURE, °C.	OBSERVED PER CENT OF SUGAR													
	0	5	10	15	20	25	30	35	40	45	50	55	60	70
	Subtract—													
0	0.30	0.49	0.65	0.77	0.89	0.99	1.08	1.16	1.24	1.31	1.37	1.41	1.44	1.49
5	0.36	0.47	0.56	0.65	0.73	0.80	0.86	0.91	0.97	1.01	1.05	1.08	1.10	1.14
10	0.32	0.38	0.43	0.48	0.52	0.57	0.60	0.64	0.67	0.70	0.72	0.74	0.75	0.77
11	0.31	0.35	0.40	0.44	0.48	0.51	0.55	0.58	0.60	0.63	0.65	0.66	0.68	0.70
12	0.29	0.32	0.36	0.40	0.43	0.46	0.50	0.52	0.54	0.56	0.58	0.59	0.60	0.62
13	0.26	0.29	0.32	0.35	0.38	0.41	0.44	0.46	0.48	0.49	0.51	0.52	0.53	0.55
14	0.24	0.26	0.29	0.31	0.34	0.36	0.38	0.40	0.41	0.42	0.44	0.45	0.46	0.47
15	0.20	0.22	0.24	0.26	0.28	0.30	0.32	0.33	0.34	0.36	0.36	0.37	0.38	0.39
16	0.17	0.18	0.20	0.22	0.23	0.25	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32
17	0.13	0.14	0.15	0.16	0.18	0.19	0.20	0.20	0.21	0.21	0.22	0.23	0.23	0.24
17.5	0.11	0.12	0.12	0.14	0.15	0.16	0.16	0.17	0.17	0.18	0.18	0.19	0.19	0.20
18	0.09	0.10	0.10	0.11	0.12	0.13	0.13	0.14	0.14	0.14	0.15	0.15	0.15	0.16
19	0.05	0.05	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08
	Add—													
21	0.04	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.09
22	0.10	0.10	0.11	0.12	0.12	0.13	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.16
23	0.16	0.16	0.17	0.17	0.19	0.20	0.21	0.21	0.22	0.23	0.24	0.24	0.24	0.24
24	0.21	0.22	0.23	0.24	0.26	0.27	0.28	0.29	0.30	0.31	0.32	0.32	0.32	0.32
25	0.27	0.28	0.30	0.31	0.32	0.34	0.35	0.36	0.38	0.38	0.39	0.39	0.40	0.39
26	0.33	0.34	0.36	0.37	0.40	0.40	0.42	0.44	0.46	0.47	0.47	0.48	0.48	0.48
27	0.40	0.41	0.42	0.44	0.46	0.48	0.50	0.52	0.54	0.54	0.55	0.56	0.56	0.56
28	0.46	0.47	0.49	0.51	0.54	0.56	0.58	0.60	0.61	0.62	0.63	0.64	0.64	0.64
29	0.54	0.55	0.56	0.59	0.61	0.63	0.66	0.68	0.70	0.70	0.71	0.72	0.72	0.72
30	0.61	0.62	0.63	0.66	0.68	0.71	0.73	0.76	0.78	0.78	0.79	0.80	0.80	0.81
35	0.99	1.01	1.02	1.06	1.10	1.13	1.16	1.18	1.20	1.21	1.22	1.22	1.23	1.22
40	1.42	1.45	1.47	1.51	1.54	1.57	1.60	1.62	1.64	1.65	1.65	1.65	1.66	1.65
45	1.91	1.94	1.96	2.00	2.03	2.05	2.07	2.09	2.10	2.10	2.10	2.10	2.10	2.08
50	2.46	2.48	2.50	2.53	2.56	2.57	2.58	2.59	2.59	2.58	2.58	2.57	2.56	2.52
55	3.05	3.07	3.09	3.12	3.12	3.12	3.12	3.11	3.10	3.08	3.07	3.05	3.03	2.97
60	3.69	3.72	3.73	3.73	3.72	3.70	3.67	3.65	3.62	3.60	3.57	3.54	3.50	3.43
65	4.4	4.4	4.4	4.4	4.4	4.4	4.3	4.2	4.2	4.1	4.1	4.0	4.0	3.9
70	5.1	5.1	5.1	5.0	5.0	5.0	4.9	4.8	4.8	4.7	4.7	4.6	4.6	4.4
75	6.1	6.0	6.0	5.9	5.8	5.8	5.7	5.6	5.5	5.4	5.4	5.3	5.2	5.0
80	7.1	7.0	7.0	6.9	6.8	6.7	6.6	6.4	6.3	6.2	6.1	6.0	5.9	5.6

The above table may also be used with instruments that are standard at 17.5°C., as follows: Find the correction for reducing 20°C. in the usual way, and to this add the correction at 17.5°C. with the sign changed; i. e., regarded as positive.

For example, if the instrument reads 20 per cent at 24°C., the correction to 17.5°C. is $+0.26+0.15=0.41$, and the true per cent of sugar is 20.41. If it reads 20 per cent at 18°C., the correction to 17.5°C. is $-0.12+0.15=+0.03$, and the true per cent of sugar is 20.03. If it reads 20 at 15°C., the correction is $-0.28+0.15=-0.13$, and the true per cent of sugar is 19.87.

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT $\frac{20° C.}{4°}$	SPECIFIC GRAVITY AT $\frac{20° C.}{20°}$	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT $\frac{20° C.}{4°}$	SPECIFIC GRAVITY AT $\frac{20° C.}{20°}$	DEGREES BAUMÉ (MODULUS 145)
0.0	0.99823	1.00000	0.00	4.5	1.01586	1.01766	2.52
0.1	0.99862	1.00039	0.06	4.6	1.01626	1.01806	2.57
0.2	0.99901	1.00078	0.11	4.7	1.01666	1.01846	2.63
0.3	0.99940	1.00117	0.17	4.8	1.01706	1.01886	2.68
0.4	0.99979	1.00155	0.22	4.9	1.01746	1.01926	2.74
0.5	1.00017	1.00194	0.28	5.0	1.01785	1.01965	2.79
0.6	1.00056	1.00233	0.34	5.1	1.01825	1.02005	2.85
0.7	1.00095	1.00272	0.39	5.2	1.01865	1.02045	2.91
0.8	1.00134	1.00311	0.45	5.3	1.01905	1.02085	2.96
0.9	1.00173	1.00350	0.51	5.4	1.01945	1.02125	3.02
1.0	1.00212	1.00389	0.56	5.5	1.01985	1.02165	3.07
1.1	1.00251	1.00428	0.62	5.6	1.02025	1.02206	3.13
1.2	1.00290	1.00467	0.67	5.7	1.02065	1.02246	3.18
1.3	1.00329	1.00506	0.73	5.8	1.02105	1.02286	3.24
1.4	1.00368	1.00545	0.79	5.9	1.02145	1.02321	3.30
1.5	1.00406	1.00584	0.84	6.0	1.02186	1.02366	3.35
1.6	1.00445	1.00623	0.90	6.1	1.02226	1.02407	3.41
1.7	1.00484	1.00662	0.95	6.2	1.02266	1.02447	3.46
1.8	1.00523	1.00701	1.01	6.3	1.02306	1.02487	3.52
1.9	1.00562	1.00740	1.07	6.4	1.02346	1.02527	3.57
2.0	1.00602	1.00779	1.12	6.5	1.02387	1.02568	3.63
2.1	1.00641	1.00818	1.18	6.6	1.02427	1.02608	3.69
2.2	1.00680	1.00858	1.23	6.7	1.02467	1.02648	3.74
2.3	1.00719	1.00897	1.29	6.8	1.02508	1.02689	3.80
2.4	1.00759	1.00936	1.34	6.9	1.02548	1.02729	3.85
2.5	1.00797	1.00976	1.40	7.0	1.02588	1.02770	3.91
2.6	1.00836	1.01015	1.46	7.1	1.02629	1.02810	3.96
2.7	1.00876	1.01054	1.51	7.2	1.02669	1.02851	4.02
2.8	1.00915	1.01093	1.57	7.3	1.02710	1.02892	4.08
2.9	1.00954	1.01133	1.62	7.4	1.02750	1.02932	4.13
3.0	1.00993	1.01172	1.68	7.5	1.02791	1.02973	4.19
3.1	1.01033	1.01211	1.74	7.6	1.02832	1.03013	4.24
3.2	1.01072	1.01251	1.79	7.7	1.02872	1.03054	4.30
3.3	1.01112	1.01290	1.85	7.8	1.02913	1.03095	4.35
3.4	1.01151	1.01330	1.90	7.9	1.02954	1.03136	4.41
3.5	1.01190	1.01369	1.96	8.0	1.02994	1.03176	4.46
3.6	1.01230	1.01409	2.02	8.1	1.03035	1.03217	4.52
3.7	1.01269	1.01448	2.07	8.2	1.03076	1.03258	4.58
3.8	1.01309	1.01488	2.13	8.3	1.03116	1.03299	4.63
3.9	1.01348	1.01528	2.18	8.4	1.03157	1.03340	4.69
4.0	1.01388	1.01567	2.24	8.5	1.03198	1.03381	4.74
4.1	1.01428	1.01607	2.29	8.6	1.03239	1.03422	4.80
4.2	1.01467	1.01647	2.35	8.7	1.03280	1.03463	4.85
4.3	1.01507	1.01687	2.40	8.8	1.03321	1.03504	4.91
4.4	1.01547	1.01726	2.46	8.9	1.03362	1.03545	4.96

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)
9.0	1.03403	1.03586	5.02	13.5	1.05278	1.05464	7.51
9.1	1.03444	1.03627	5.07	13.6	1.05320	1.05506	7.57
9.2	1.03485	1.03668	5.13	13.7	1.05363	1.05549	7.62
9.3	1.03526	1.03709	5.19	13.8	1.05405	1.05591	7.68
9.4	1.03567	1.03750	5.24	13.9	1.05448	1.05634	7.73
9.5	1.03608	1.03792	5.30	14.0	1.05490	1.05677	7.79
9.6	1.03649	1.03833	5.35	14.1	1.05532	1.05719	7.84
9.7	1.03691	1.03874	5.41	14.2	1.05575	1.05762	7.90
9.8	1.03732	1.03915	5.46	14.3	1.05618	1.05804	7.96
9.9	1.03773	1.03957	5.52	14.4	1.05660	1.05847	8.01
10.0	1.03814	1.03998	5.57	14.5	1.05703	1.05890	8.07
10.1	1.03856	1.04039	5.63	14.6	1.05746	1.05933	8.12
10.2	1.03897	1.04081	5.68	14.7	1.05788	1.05975	8.18
10.3	1.03938	1.04122	5.74	14.8	1.05831	1.06018	8.23
10.4	1.03980	1.04164	5.80	14.9	1.05874	1.06061	8.29
10.5	1.04021	1.04205	5.85	15.0	1.05916	1.06104	8.34
10.6	1.04063	1.04247	5.91	15.1	1.05959	1.06147	8.40
10.7	1.04104	1.04288	5.96	15.2	1.06002	1.06190	8.45
10.8	1.04146	1.04330	6.02	15.3	1.06045	1.06233	8.51
10.9	1.04187	1.04371	6.07	15.4	1.06088	1.06276	8.56
11.0	1.04229	1.04413	6.13	15.5	1.06131	1.06319	8.62
11.1	1.04270	1.04455	6.18	15.6	1.06174	1.06362	8.67
11.2	1.04312	1.04497	6.24	15.7	1.06217	1.06405	8.73
11.3	1.04354	1.04538	6.30	15.8	1.06260	1.06448	8.78
11.4	1.04395	1.04580	6.35	15.9	1.06303	1.06491	8.84
11.5	1.04437	1.04622	6.41	16.0	1.06346	1.06534	8.89
11.6	1.04479	1.04664	6.46	16.1	1.06389	1.06577	8.95
11.7	1.04521	1.04706	6.52	16.2	1.06432	1.06621	9.00
11.8	1.04562	1.04747	6.57	16.3	1.06476	1.06664	9.06
11.9	1.04604	1.04789	6.63	16.4	1.06519	1.06707	9.11
12.0	1.04646	1.04831	6.68	16.5	1.06562	1.06751	9.17
12.1	1.04688	1.04873	6.74	16.6	1.06605	1.06794	9.22
12.2	1.04730	1.04915	6.79	16.7	1.06649	1.06837	9.28
12.3	1.04772	1.04957	6.85	16.8	1.06692	1.06881	9.33
12.4	1.04814	1.04999	6.90	16.9	1.06736	1.06924	9.39
12.5	1.04856	1.05041	6.96	17.0	1.06779	1.06968	9.45
12.6	1.04898	1.05084	7.02	17.1	1.06822	1.07011	9.50
12.7	1.04940	1.05126	7.07	17.2	1.06866	1.07055	9.56
12.8	1.04982	1.05168	7.13	17.3	1.06909	1.07098	9.61
12.9	1.05024	1.05210	7.18	17.4	1.06953	1.07142	9.67
13.0	1.05066	1.05252	7.24	17.5	1.06996	1.07186	9.72
13.1	1.05109	1.05295	7.29	17.6	1.07040	1.07229	9.78
13.2	1.05151	1.05337	7.35	17.7	1.07084	1.07273	9.83
13.3	1.05193	1.05379	7.40	17.8	1.07127	1.07317	9.89
13.4	1.05236	1.05422	7.46	17.9	1.07171	1.07361	9.94

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C./4°	SPECIFIC GRAVITY AT 20° C./20°	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C./4°	SPECIFIC GRAVITY AT 20° C./20°	DEGREES BAUMÉ (MODULUS 145)
18.0	1.07215	1.07404	10.00	22.5	1.09216	1.09409	12.47
18.1	1.07258	1.07448	10.05	22.6	1.09261	1.09454	12.52
18.2	1.07302	1.07492	10.11	22.7	1.09306	1.09499	12.58
18.3	1.07346	1.07536	10.16	22.8	1.09351	1.09545	12.63
18.4	1.07390	1.07580	10.22	22.9	1.09397	1.09590	12.69
18.5	1.07434	1.07624	10.27	23.0	1.09442	1.09636	12.74
18.6	1.07478	1.07668	10.33	23.1	1.09487	1.09681	12.80
18.7	1.07522	1.07712	10.38	23.2	1.09533	1.09727	12.85
18.8	1.07566	1.07756	10.44	23.3	1.09578	1.09772	12.91
18.9	1.07610	1.07800	10.49	23.4	1.09624	1.09818	12.96
19.0	1.07654	1.07844	10.55	23.5	1.09669	1.09863	13.02
19.1	1.07698	1.07888	10.60	23.6	1.09715	1.09909	13.07
19.2	1.07742	1.07932	10.66	23.7	1.09760	1.09954	13.13
19.3	1.07786	1.07977	10.71	23.8	1.09806	1.10000	13.18
19.4	1.07830	1.08021	10.77	23.9	1.09851	1.10046	13.24
19.5	1.07874	1.08065	10.82	24.0	1.09897	1.10092	13.29
19.6	1.07919	1.08110	10.88	24.1	1.09943	1.10137	13.35
19.7	1.07963	1.08154	10.93	24.2	1.09989	1.10183	13.40
19.8	1.08007	1.08198	10.99	24.3	1.10034	1.10229	13.46
19.9	1.08052	1.08243	11.04	24.4	1.10080	1.10275	13.51
20.0	1.08096	1.08287	11.10	24.5	1.10126	1.10321	13.57
20.1	1.08140	1.08332	11.15	24.6	1.10172	1.10367	13.62
20.2	1.08185	1.08376	11.21	24.7	1.10218	1.10413	13.67
20.3	1.08229	1.08421	11.26	24.8	1.10264	1.10459	13.73
20.4	1.08274	1.08465	11.32	24.9	1.10310	1.10505	13.78
20.5	1.08318	1.08510	11.37	25.0	1.10356	1.10551	13.84
20.6	1.08363	1.08554	11.43	25.1	1.10402	1.10597	13.89
20.7	1.08407	1.08599	11.48	25.2	1.10448	1.10643	13.95
20.8	1.08452	1.08644	11.54	25.3	1.10494	1.10689	14.00
20.9	1.08497	1.08689	11.59	25.4	1.10540	1.10736	14.06
21.0	1.08541	1.08733	11.65	25.5	1.10586	1.10782	14.11
21.1	1.08586	1.08778	11.70	25.6	1.10632	1.10828	14.17
21.2	1.08631	1.08823	11.76	25.7	1.10679	1.10874	14.22
21.3	1.08676	1.08868	11.81	25.8	1.10725	1.10921	14.28
21.4	1.08720	1.08913	11.87	25.9	1.10771	1.10967	14.33
21.5	1.08765	1.08958	11.92	26.0	1.10818	1.11014	14.39
21.6	1.08810	1.09003	11.98	26.1	1.10864	1.11060	14.44
21.7	1.08855	1.09048	12.03	26.2	1.10910	1.11106	14.49
21.8	1.08900	1.09093	12.09	26.3	1.10957	1.11153	14.55
21.9	1.08945	1.09138	12.14	26.4	1.11003	1.11200	14.60
22.0	1.08990	1.09183	12.20	26.5	1.11050	1.11246	14.66
22.1	1.09035	1.09228	12.25	26.6	1.11096	1.11293	14.71
22.2	1.09080	1.09273	12.31	26.7	1.11143	1.11339	14.77
22.3	1.09125	1.09318	12.36	26.8	1.11190	1.11386	14.82
22.4	1.09170	1.09364	12.42	26.9	1.11236	1.11433	14.88

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C./4°	SPECIFIC GRAVITY AT 20° C./20°	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C./4°	SPECIFIC GRAVITY AT 20° C./20°	DEGREES BAUMÉ (MODULUS 145)
27.0	1.11283	1.11480	14.93	31.5	1.13418	1.13619	17.38
27.1	1.11330	1.11526	14.99	31.6	1.13466	1.13667	17.43
27.2	1.11376	1.11573	15.04	31.7	1.13515	1.13715	17.49
27.3	1.11423	1.11620	15.09	31.8	1.13563	1.13764	17.54
27.4	1.11470	1.11667	15.15	31.9	1.13611	1.13812	17.60
27.5	1.11517	1.11714	15.20	32.0	1.13660	1.13861	17.65
27.6	1.11564	1.11761	15.26	32.1	1.13708	1.13909	17.70
27.7	1.11610	1.11808	15.31	32.2	1.13756	1.13958	17.76
27.8	1.11657	1.11855	15.37	32.3	1.13805	1.14006	17.81
27.9	1.11704	1.11902	15.42	32.4	1.13853	1.14055	17.87
28.0	1.11751	1.11949	15.48	32.5	1.13902	1.14103	17.92
28.1	1.11798	1.11996	15.53	32.6	1.13951	1.14152	17.98
28.2	1.11845	1.12043	15.59	32.7	1.13999	1.14201	18.03
28.3	1.11892	1.12090	15.64	32.8	1.14048	1.14250	18.08
28.4	1.11940	1.12138	15.69	32.9	1.14097	1.14298	18.14
28.5	1.11987	1.12185	15.75	33.0	1.14145	1.14347	18.19
28.6	1.12034	1.12232	15.80	33.1	1.14194	1.14396	18.25
28.7	1.12081	1.12280	15.86	33.2	1.14243	1.14445	18.30
28.8	1.12128	1.12327	15.91	33.3	1.14292	1.14494	18.36
28.9	1.12176	1.12374	15.97	33.4	1.14340	1.14543	18.41
29.0	1.12223	1.12422	16.02	33.5	1.14389	1.14592	18.46
29.1	1.12270	1.12469	16.08	33.6	1.14438	1.14641	18.52
29.2	1.12318	1.12517	16.13	33.7	1.14487	1.14690	18.57
29.3	1.12365	1.12564	16.18	33.8	1.14536	1.14739	18.63
29.4	1.12413	1.12612	16.24	33.9	1.14585	1.14788	18.68
29.5	1.12460	1.12659	16.29	34.0	1.14634	1.14837	18.73
29.6	1.12508	1.12707	16.35	34.1	1.14684	1.14886	18.79
29.7	1.12556	1.12755	16.40	34.2	1.14733	1.14936	18.84
29.8	1.12603	1.12802	16.46	34.3	1.14782	1.14985	18.90
29.9	1.12651	1.12850	16.51	34.4	1.14831	1.15034	18.95
30.0	1.12698	1.12898	16.57	34.5	1.14880	1.15084	19.00
30.1	1.12746	1.12946	16.62	34.6	1.14930	1.15133	19.06
30.2	1.12794	1.12993	16.67	34.7	1.14979	1.15183	19.11
30.3	1.12842	1.13041	16.73	34.8	1.15029	1.15232	19.17
30.4	1.12890	1.13089	16.78	34.9	1.15078	1.15282	19.22
30.5	1.12937	1.13137	16.84	35.0	1.15128	1.15331	19.28
30.6	1.12985	1.13185	16.89	35.1	1.15177	1.15381	19.33
30.7	1.13033	1.13233	16.95	35.2	1.15226	1.15430	19.38
30.8	1.13081	1.13281	17.00	35.3	1.15276	1.15480	19.44
30.9	1.13129	1.13329	17.05	35.4	1.15326	1.15530	19.49
31.0	1.13177	1.13378	17.11	35.5	1.15375	1.15579	19.55
31.1	1.13225	1.13426	17.16	35.6	1.15425	1.15629	19.60
31.2	1.13274	1.13474	17.22	35.7	1.15475	1.15679	19.65
31.3	1.13322	1.13522	17.27	35.8	1.15524	1.15729	19.71
31.4	1.13370	1.13570	17.33	35.9	1.15574	1.15778	19.76

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT $\frac{20^\circ\text{ C.}}{4^\circ}$	SPECIFIC GRAVITY AT $\frac{20^\circ\text{ C.}}{20^\circ}$	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT $\frac{20^\circ\text{ C.}}{4^\circ}$	SPECIFIC GRAVITY AT $\frac{20^\circ\text{ C.}}{20^\circ}$	DEGREES BAUMÉ (MODULUS 145)
36.0	1.15624	1.15828	19.81	40.5	1.17901	1.18110	22.23
36.1	1.15674	1.15878	19.87	40.6	1.17953	1.18162	22.29
36.2	1.15724	1.15928	19.92	40.7	1.18004	1.18213	22.34
36.3	1.15773	1.15978	19.98	40.8	1.18056	1.18265	22.39
36.4	1.15823	1.16028	20.03	40.9	1.18108	1.18316	22.45
36.5	1.15873	1.16078	20.08	41.0	1.18159	1.18368	22.50
36.6	1.15923	1.16128	20.14	41.1	1.18211	1.18420	22.55
36.7	1.15973	1.16178	20.19	41.2	1.18262	1.18472	22.61
36.8	1.16023	1.16228	20.25	41.3	1.18314	1.18524	22.66
36.9	1.16073	1.16279	20.30	41.4	1.18366	1.18575	22.72
37.0	1.16124	1.16329	20.35	41.5	1.18418	1.18627	22.77
37.1	1.16174	1.16379	20.41	41.6	1.18470	1.18679	22.82
37.2	1.16224	1.16430	20.46	41.7	1.18522	1.18731	22.88
37.3	1.16274	1.16480	20.52	41.8	1.18573	1.18783	22.93
37.4	1.16324	1.16530	20.57	41.9	1.18625	1.18835	22.98
37.5	1.16375	1.16581	20.62	42.0	1.18677	1.18887	23.04
37.6	1.16425	1.16631	20.68	42.1	1.18729	1.18939	23.09
37.7	1.16476	1.16682	20.73	42.2	1.18781	1.18992	23.14
37.8	1.16526	1.16732	20.78	42.3	1.18834	1.19044	23.20
37.9	1.16576	1.16783	20.84	42.4	1.18886	1,19096	23.25
38.0	1.16627	1.16833	20.89	42.5	1.18938	1.19148	23.30
38.1	1.16678	1.16884	20.94	42.6	1.18990	1.19201	23.36
38.2	1.16728	1.16934	21.00	42.7	1.19042	1.19253	23.41
38.3	1.16779	1.16985	21.05	42.8	1.19095	1.19305	23.46
38.4	1.16829	1.17036	21.11	42.9	1.19147	1.19358	23.52
38.5	1.16880	1.17087	21.16	43.0	1.19199	1.19410	23.57
38.6	1.16931	1.17138	21.21	43.1	1.19252	1.19463	23.62
38.7	1.16982	1.17188	21.27	43.2	1.19304	1.19515	23.68
38.8	1.17032	1.17239	21.32	43.3	1.19356	1.19568	23.73
38.9	1.17083	1.17290	21.38	43.4	1.19409	1.19620	23.78
39.0	1.17134	1.17341	21.43	43.5	1.19462	1.19673	23.84
39.1	1.17183	1.17392	21.48	43.6	1.19514	1.19726	23.89
39.2	1.17236	1.17443	21.54	43.7	1.19567	1.19778	23.94
39.3	1.17287	1.17494	21.59	43.8	1.19619	1.19831	24.00
39.4	1.17338	1.17545	21.64	43.9	1.19672	1.19884	24.05
39.5	1.17389	1.17596	21.70	44.0	1.19725	1.19936	24.10
39.6	1.17440	1.17648	21.75	44.1	1.19778	1.19989	24.16
39.7	1.17491	1.17699	21.80	44.2	1.19830	1.20042	24.21
39.8	1.17542	1.17750	21.86	44.3	1.19883	1.20095	24.26
39.9	1.17594	1.17802	21.91	44.4	1.19936	1.20148	24.32
40.0	1.17645	1.17853	21.97	44.5	1.19989	1.20201	24.37
40.1	1.17696	1.17904	22.02	44.6	1.20042	1.20254	24.42
40.2	1.17747	1.17956	22.07	44.7	1.20095	1.20307	24.48
40.3	1.17799	1.18007	22.13	44.8	1.20148	1.20360	24.53
40.4	1.17850	1.18058	22.18	44.9	1.20201	1.20414	24.58

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)
45.0	1.20254	1.20467	24.63	49.5	1.22682	1.22899	27.02
45.1	1.20307	1.20520	24.69	49.6	1.22737	1.22954	27.07
45.2	1.20360	1.20573	24.74	49.7	1.22792	1.23009	27.12
45.3	1.20414	1.20627	24.79	49.8	1.22847	1.23064	27.18
45.4	1.20467	1.20680	24.85	49.9	1.22902	1.23119	27.23
45.5	1.20520	1.20733	24.90	50.0	1.22957	1.23174	27.28
45.6	1.20573	1.20787	24.95	50.1	1.23012	1.23229	27.33
45.7	1.20627	1.20840	25.01	50.2	1.23067	1.23284	27.39
45.8	1.20680	1.20894	25.06	50.3	1.23122	1.23340	27.44
45.9	1.20734	1.20947	25.11	50.4	1.23177	1.23395	27.49
46.0	1.20787	1.21001	25.17	50.5	1.23232	1.23450	27.54
46.1	1.20840	1.21054	25.22	50.6	1.23287	1.23506	27.60
46.2	1.20894	1.21108	25.27	50.7	1.23343	1.23561	27.65
46.3	1.20948	1.21162	25.32	50.8	1.23398	1.23616	27.70
46.4	1.21001	1.21215	25.38	50.9	1.23453	1.23672	27.75
46.5	1.21055	1.21269	25.43	51.0	1.23508	1.23727	27.81
46.6	1.21109	1.21323	25.48	51.1	1.23564	1.23782	27.86
46.7	1.21162	1.21377	25.54	51.2	1.23619	1.23838	27.91
46.8	1.21216	1.21431	25.59	51.3	1.23675	1.23894	27.96
46.9	1.21270	1.21484	25.64	51.4	1.23730	1.23949	28.02
47.0	1.21324	1.21538	25.70	51.5	1.23786	1.24005	28.07
47.1	1.21378	1.21592	25.75	51.6	1.23841	1.24060	28.12
47.2	1.21432	1.21646	25.80	51.7	1.23897	1.24116	28.17
47.3	1.21486	1.21700	25.86	51.8	1.23953	1.24172	28.23
47.4	1.21540	1.21755	25.91	51.9	1.24008	1.24228	28.28
47.5	1.21594	1.21809	25.96	52.0	1.24064	1.24284	28.33
47.6	1.21648	1.21863	26.01	52.1	1.24120	1.24339	28.38
47.7	1.21702	1.21917	26.07	52.2	1.24176	1.24395	28.44
47.8	1.21756	1.21971	26.12	52.3	1.24232	1.24451	28.49
47.9	1.21810	1.22026	26.17	52.4	1.24287	1.34507	28.54
48.0	1.21864	1.22080	26.23	52.5	1.24343	1.24563	28.59
48.1	1.21918	1.22134	26.28	52.6	1.24399	1.24619	28.65
48.2	1.21973	1.22189	26.33	52.7	1.24455	1.24675	28.70
48.3	1.22027	1.22243	26.38	52.8	1.24511	1.24731	28.75
48.4	1.22082	1.22298	26.44	52.9	1.24567	1.24788	28.80
48.5	1.22136	1.22352	26.49	53.0	1.24623	1.24844	28.86
48.6	1.22190	1.22406	26.54	53.1	1.24680	1.24900	28.91
48.7	1.22245	1.22461	26.59	53.2	1.24736	1.24956	28.96
48.8	1.22300	1.22516	26.65	53.3	1.24792	1.25013	29.01
48.9	1.22354	1.22570	26.70	53.4	1.24848	1.25069	29.06
49.0	1.22409	1.22625	26.75	53.5	1.24905	1.25126	29.12
49.1	1.22463	1.22680	26.81	53.6	1.24961	1.25182	29.17
49.2	1.22518	1.22735	26.86	53.7	1.25017	1.25238	29.22
49.3	1.22573	1.22789	26.91	53.8	1.25074	1.25295	29.27
49.4	1.22627	1.22844	26.96	53.9	1.25130	1.25351	29.32

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT $\frac{20^\circ\text{ C.}}{4^\circ}$	SPECIFIC GRAVITY AT $\frac{20^\circ\text{ C.}}{20^\circ}$	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT $\frac{20^\circ\text{ C.}}{4^\circ}$	SPECIFIC GRAVITY AT $\frac{20^\circ\text{ C.}}{20^\circ}$	DEGREES BAUMÉ (MODULUS 145)
54.0	1.25187	1.25408	29.38	58.5	1.27768	1.27994	31.71
54.1	1.25243	1.25465	29.43	58.6	1.27826	1.28052	31.76
54.2	1.25300	1.25521	29.48	58.7	1.27884	1.28111	31.82
54.3	1.25356	1.25578	29.53	58.8	1.27943	1.28169	31.87
54.4	1.25413	1.25635	29.59	58.9	1.28001	1.28228	31.92
54.5	1.25470	1.25692	29.64	59.0	1.28060	1.28286	31.97
54.6	1.25526	1.25748	29.69	59.1	1.28118	1.28345	32.02
54.7	1.25583	1.25805	29.74	59.2	1.28176	1.28404	32.07
54.8	1.25640	1.25862	29.80	59.3	1.28235	1.28462	32.13
54.9	1.25697	1.25919	29.85	59.4	1.28294	1.28520	32.18
55.0	1.25754	1.25976	29.90	59.5	1.28352	1.28579	32.23
55.1	1.25810	1.26033	29.95	59.6	1.28411	1.28638	32.28
55.2	1.25867	1.26090	30.00	59.7	1.28469	1.28697	32.33
55.3	1.25924	1.26147	30.06	59.8	1.28528	1.28755	32.38
55.4	1.25982	1.26204	30.11	59.9	1.28587	1.28814	32.43
55.5	1.26039	1.26261	30.16	60.0	1.28646	1.28873	32.49
55.6	1.26096	1.26319	30.21	60.1	1.28704	1.28932	32.54
55.7	1.26153	1.26376	30.26	60.2	1.28763	1.28991	32.59
55.8	1.26210	1.26433	30.32	60.3	1.28822	1.29050	32.64
55.9	1.26267	1.26490	30.37	60.4	1.28881	1.29109	32.69
56.0	1.26324	1.26548	30.42	60.5	1.28940	1.29168	32.74
56.1	1.26382	1.26605	30.47	60.6	1.28999	1.29227	32.79
56.2	1.26439	1.26663	30.52	60.7	1.29058	1.29286	32.85
56.3	1.26496	1.26720	30.57	60.8	1.29117	1.29346	32.90
56.4	1.26554	1.26778	30.63	60.9	1.29176	1.29405	32.95
56.5	1.26611	1.26835	30.68	61.0	1.29235	1.29464	33.00
56.6	1.26669	1.26893	30.73	61.1	1.29295	1.29523	33.05
56.7	1.26726	1.26950	30.78	61.2	1.29354	1.29583	33.10
56.8	1.26784	1.27008	30.83	61.3	1.29413	1.29642	33.15
56.9	1.26841	1.27066	30.89	61.4	1.29472	1.29701	33.20
57.0	1.26899	1.27123	30.94	61.5	1.29532	1.29761	33.26
57.1	1.26956	1.27181	30.99	61.6	1.29591	1.29820	33.31
57.2	1.27014	1.27239	31.04	61.7	1.29651	1.29880	33.36
57.3	1.27072	1.27297	31.09	61.8	1.29710	1.29940	33.41
57.4	1.27130	1.27355	31.15	61.9	1.29770	1.29999	33.46
57.5	1.27188	1.27413	31.20	62.0	1.29829	1.30059	33.51
57.6	1.27246	1.27471	31.25	62.1	1.29889	1.30118	33.56
57.7	1.27304	1.27529	31.30	62.2	1.29948	1.30178	33.61
57.8	1.27361	1.27587	31.35	62.3	1.30008	1.30238	33.67
57.9	1.27419	1.27645	31.40	62.4	1.30068	1.30298	33.72
58.0	1.27477	1.27703	31.46	62.5	1.30127	1.30358	33.77
58.1	1.27535	1.27761	31.51	62.6	1.30187	1.30418	33.82
58.2	1.27594	1.27819	31.56	62.7	1.30247	1.30477	33.87
58.3	1.27652	1.27878	31.61	62.8	1.30307	1.30537	33.92
58.4	1.27710	1.27936	31.66	62.9	1.30367	1.30597	33.97

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions*.—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)
63.0	1.30427	1.30657	34.02	67.5	1.33163	1.33399	36.30
63.1	1.30487	1.30718	34.07	67.6	1.33225	1.33460	36.35
63.2	1.30547	1.30778	34.12	67.7	1.33287	1.33523	36.40
63.3	1.30607	1.30838	34.18	67.8	1.33348	1.33584	36.45
63.4	1.30667	1.30898	34.23	67.9	1.33410	1.33646	36.50
63.5	1.30727	1.30958	34.28	68.0	1.33472	1.33708	36.55
63.6	1.30787	1.31019	34.33	68.1	1.33534	1.33770	36.61
63.7	1.30848	1.31079	34.38	68.2	1.33596	1.33832	36.66
63.8	1.30908	1.31139	34.43	68.3	1.33658	1.33894	36.71
63.9	1.30968	1.31200	34.48	68.4	1.33720	1.33957	36.76
64.0	1.31028	1.31260	34.53	68.5	1.33782	1.34019	36.81
64.1	1.31088	1.31320	34.58	68.6	1.33844	1.34081	36.86
64.2	1.31149	1.31381	34.63	68.7	1.33906	1.34143	36.91
64.3	1.31209	1.31441	34.68	68.8	1.33968	1.34205	36.96
64.4	1.31270	1.31502	34.74	68.9	1.34031	1.34268	37.01
64.5	1.31330	1.31563	34.79	69.0	1.34093	1.34330	37.06
64.6	1.31391	1.31623	34.84	69.1	1.34155	1.34392	37.11
64.7	1.31452	1.31684	34.89	69.2	1.34217	1.34455	37.16
64.8	1.31512	1.31745	34.94	69.3	1.34280	1.34517	37.21
64.9	1.31573	1.31806	34.99	69.4	1.34342	1.34580	37.26
65.0	1.31633	1.31866	35.04	69.5	1.34405	1.34642	37.31
65.1	1.31694	1.31927	35.09	69.6	1.34467	1.34705	37.36
65.2	1.31755	1.31988	35.14	69.7	1.34530	1.34768	37.41
65.3	1.31816	1.32049	35.19	69.8	1.34592	1.34830	37.46
65.4	1.31877	1.32110	35.24	69.9	1.34655	1.34893	37.51
65.5	1.31937	1.32171	35.29	70.0	1.34717	1.34956	37.56
65.6	1.31998	1.32232	35.34	70.1	1.34780	1.35019	37.61
65.7	1.32059	1.32293	35.39	70.2	1.34843	1.35081	37.66
65.8	1.32120	1.32354	35.45	70.3	1.34906	1.35144	37.71
65.9	1.32181	1.32415	35.50	70.4	1.34968	1.35207	37.76
66.0	1.32242	1.32476	35.55	70.5	1.35031	1.35270	37.81
66.1	1.32304	1.32538	35.60	70.6	1.35094	1.35333	37.86
66.2	1.32365	1.32599	35.65	70.7	1.35157	1.35396	37.91
66.3	1.32426	1.32660	35.70	70.8	1.35220	1.35459	37.96
66.4	1.32487	1.32722	35.75	70.9	1.35283	1.35522	38.01
66.5	1.32548	1.32783	35.80	71.0	1.35346	1.35585	38.06
66.6	1.32610	1.32844	35.85	71.1	1.35409	1.35648	38.11
66.7	1.32671	1.32906	35.90	71.2	1.35472	1.35711	38.16
66.8	1.32732	1.32967	35.95	71.3	1.35535	1.35775	38.21
66.9	1.32794	1.33029	36.00	71.4	1.35598	1.35838	38.26
67.0	1.32855	1.33090	36.05	71.5	1.35661	1.35901	38.30
67.1	1.32917	1.33152	36.10	71.6	1.35724	1.35964	38.35
67.2	1.32978	1.33214	36.15	71.7	1.35788	1.36028	38.40
67.3	1.33040	1.33275	36.20	71.8	1.35851	1.36091	38.45
67.4	1.33102	1.33337	36.25	71.9	1.35914	1.36155	38.50

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT $\frac{20^\circ \text{ C.}}{4^\circ}$	SPECIFIC GRAVITY AT $\frac{20^\circ \text{ C.}}{20^\circ}$	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT $\frac{20^\circ \text{ C.}}{4^\circ}$	SPECIFIC GRAVITY AT $\frac{20^\circ \text{ C.}}{20^\circ}$	DEGREES BAUMÉ (MODULUS 145)
72.0	1.35978	1.36218	38.55	76.5	1.38870	1.39115	40.77
72.1	1.36041	1.36282	38.60	76.6	1.38935	1.39180	40.82
72.2	1.36105	1.36346	38.65	76.7	1.39000	1.39246	40.87
72.3	1.36168	1.36409	38.70	76.8	1.39065	1.39311	40.92
72.4	1.36232	1.36473	38.75	76.9	1.39130	1.39376	40.97
72.5	1.36295	1.36536	38.80	77.0	1.39196	1.39442	41.01
72.6	1.36359	1.36600	38.85	77.1	1.39261	1.39507	41.06
72.7	1.36423	1.36664	38.90	77.2	1.39326	1.39573	41.11
72.8	1.36486	1.36728	38.95	77.3	1.39392	1.39638	41.16
72.9	1.36550	1.36792	39.00	77.4	1.39457	1.39704	41.21
73.0	1.36614	1.36856	39.05	77.5	1.39523	1.39769	41.26
73.1	1.36678	1.36919	39.10	77.6	1.39588	1.39835	41.31
73.2	1.36742	1.36983	39.15	77.7	1.39654	1.39901	41.36
73.3	1.36805	1.37047	39.20	77.8	1.39719	1.39966	41.40
73.4	1.36869	1.37111	39.25	77.9	1.39785	1.40032	41.45
73.5	1.36933	1.37176	39.30	78.0	1.39850	1.40098	41.50
73.6	1.36997	1.37240	39.35	78.1	1.39916	1.40164	41.55
73.7	1.37061	1.37304	39.39	78.2	1.39982	1.40230	41.60
73.8	1.37125	1.37368	39.44	78.3	1.40048	1.40295	41.65
73.9	1.37189	1.37432	39.49	78.4	1.40113	1.40361	41.70
74.0	1.37254	1.37496	39.54	78.5	1.40179	1.40427	41.74
74.1	1.37318	1.37561	39.59	78.6	1.40245	1.40493	41.79
74.2	1.37382	1.37625	39.64	78.7	1.40311	1.40559	41.84
74.3	1.37446	1.37689	39.69	78.8	1.40377	1.40625	41.89
74.4	1.37510	1.37754	39.74	78.9	1.40443	1.40691	41.94
74.5	1.37575	1.37818	39.79	79.0	1.40509	1.40758	41.99
74.6	1.37639	1.37883	39.84	79.1	1.40575	1.40824	42.03
74.7	1.37704	1.37947	39.89	79.2	1.40641	1.40890	42.08
74.8	1.37768	1.38012	39.94	79.3	1.40707	1.40956	42.13
74.9	1.37833	1.38076	39.99	79.4	1.40774	1.41023	42.18
75.0	1.37897	1.38141	40.03	79.5	1.40840	1.41089	42.23
75.1	1.37962	1.38206	40.08	79.6	1.40906	1.41155	42.28
75.2	1.38026	1.38270	40.13	79.7	1.40972	1.41222	42.32
75.3	1.38091	1.38335	40.18	79.8	1.41039	1.41288	42.37
75.4	1.38156	1.38400	40.23	79.9	1.41105	1.41355	42.42
75.5	1.38220	1.38465	40.28	80.0	1.41172	1.41421	42.47
75.6	1.38285	1.38530	40.33	80.1	1.41238	1.41488	42.52
75.7	1.38350	1.38595	40.38	80.2	1.41304	1.41554	42.57
75.8	1.38415	1.38660	40.43	80.3	1.41371	1.41621	42.61
75.9	1.38480	1.38725	40.48	80.4	1.41437	1.41688	42.66
76.0	1.38545	1.38790	40.53	80.5	1.41504	1.41754	42.71
76.1	1.38610	1.38855	40.57	80.6	1.41571	1.41821	42.76
76.2	1.38675	1.38920	40.62	80.7	1.41637	1.41888	42.81
76.3	1.38740	1.38985	40.67	80.8	1.41704	1.41955	42.85
76.4	1.38805	1.39050	40.72	80.9	1.41771	1.42022	42.90

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Continued.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)	DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)
81.0	1.41837	1.42088	42.95	86.0	1.45223	1.45480	45.33
81.1	1.41904	1.42155	43.00	86.1	1.45292	1.45549	45.38
81.2	1.41971	1.42222	43.05	86.2	1.45360	1.45618	45.42
81.3	1.42038	1.42289	43.10	86.3	1.45429	1.45686	45.47
81.4	1.42105	1.42356	43.14	86.4	1.45498	1.45755	45.52
81.5	1.42172	1.42423	43.19	86.5	1.45567	1.45824	45.57
81.6	1.42239	1.42490	43.24	86.6	1.45636	1.45893	45.61
81.7	1.42306	1.42558	43.29	86.7	1.45704	1.45962	45.66
81.8	1.42373	1.42625	43.33	86.8	1.45773	1.46031	45.71
81.9	1.42440	1.42692	43.38	86.9	1.45842	1.46100	45.75
82.0	1.42507	1.42759	43.43	87.0	1.45911	1.46170	45.80
82.1	1.42574	1.42827	43.48	87.1	1.45980	1.46239	45.85
82.2	1.42642	1.42894	43.53	87.2	1.46050	1.46308	45.89
82.3	1.42709	1.42961	43.57	87.3	1.46119	1.46377	45.94
82.4	1.42776	1.43029	43.62	87.4	1.46188	1.46446	45.99
82.5	1.42844	1.43096	43.67	87.5	1.46257	1.46516	46.03
82.6	1.42911	1.43164	43.72	87.6	1.46326	1.46585	46.08
82.7	1.42978	1.43231	43.77	87.7	1.46395	1.46654	46.13
82.8	1.43046	1.43298	43.81	87.8	1.46464	1.46724	46.17
82.9	1.43113	1.43366	43.86	87.9	1.46534	1.46793	46.22
83.0	1.43181	1.43434	43.91	88.0	1.46603	1.46862	46.27
83.1	1.43248	1.43502	43.96	88.1	1.46673	1.46932	46.31
83.2	1.43316	1.43569	44.00	88.2	1.46742	1.47002	46.36
83.3	1.43384	1.43637	44.05	88.3	1.46812	1.47071	46.41
83.4	1.43451	1.43705	44.10	88.4	1.46881	1.47141	46.45
83.5	1.43519	1.43773	44.15	88.5	1.46950	1.47210	46.50
83.6	1.43587	1.43841	44.19	88.6	1.47020	1.47280	46.55
83.7	1.43654	1.43908	44.24	88.7	1.47090	1.47350	46.59
83.8	1.43722	1.43976	44.29	88.8	1.47159	1.47420	46.64
83.9	1.43790	1.44044	44.34	88.9	1.47229	1.47489	46.69
84.0	1.43858	1.44112	44.38	89.0	1.47299	1.47559	46.73
84.1	1.43926	1.44180	44.43	89.1	1.47368	1.47629	46.78
84.2	1.43994	1.44249	44.48	89.2	1.47438	1.47699	46.83
84.3	1.44062	1.44317	44.53	89.3	1.47508	1.47769	46.87
84.4	1.44130	1.44385	44.57	89.4	1.47578	1.47839	46.92
84.5	1.44198	1.44453	44.62	89.5	1.47648	1.47909	46.97
84.6	1.44266	1.44521	44.67	89.6	1.47718	1.47979	47.01
84.7	1.44334	1.44590	44.72	89.7	1.47788	1.48049	47.06
84.8	1.44402	1.44658	44.76	89.8	1.47858	1.48119	47.11
84.9	1.44470	1.44726	44.81	89.9	1.47928	1.48189	47.15
85.0	1.44539	1.44794	44.86	90.0	1.47998	1.48259	47.20
85.1	1.44607	1.44863	44.91	90.1	1.48068	1.48330	47.24
85.2	1.44675	1.44931	44.95	90.2	1.48138	1.48400	47.29
85.3	1.44744	1.45000	45.00	90.3	1.48208	1.48470	47.34
85.4	1.44812	1.45068	45.05	90.4	1.48278	1.48540	47.38
85.5	1.44881	1.45137	45.09	90.5	1.48348	1.48611	47.43
85.6	1.44949	1.45205	45.14	90.6	1.48419	1.48681	47.48
85.7	1.45018	1.45274	45.19	90.7	1.48489	1.48752	47.52
85.8	1.45086	1.45343	45.24	90.8	1.48559	1.48822	47.57
85.9	1.45154	1.45411	45.28	90.9	1.48630	1.48893	47.61

10 *Degrees Brix, specific gravity, and degrees Baumé of sugar solutions.*—Concluded.

DEGREES BRIX OR PER CENT BY WEIGHT OF SUCROSE	SPECIFIC GRAVITY AT 20° C. / 4°	SPECIFIC GRAVITY AT 20° C. / 20°	DEGREES BAUMÉ (MODULUS 145)
91.0	1.48700	1.48963	47.66
91.1	1.48771	1.49034	47.71
91.2	1.48841	1.49104	47.75
91.3	1.48912	1.49175	47.80
91.4	1.48982	1.49246	47.84
91.5	1.49053	1.49316	47.89
91.6	1.49123	1.49387	47.94
91.7	1.49194	1.49458	47.98
91.8	1.49265	1.49529	48.03
91.9	1.49336	1.49600	48.08
92.0	1.49406	1.49671	48.12
92.1	1.49477	1.49741	48.17
92.2	1.49548	1.49812	48.21
92.3	1.49619	1.49883	48.26
92.4	1.49690	1.49954	48.30
92.5	1.49761	1.50026	48.35
92.6	1.49832	1.50097	48.40
92.7	1.49903	1.50168	48.44
92.8	1.49974	1.50239	48.49
92.9	1.50045	1.50310	48.53
93.0	1.50116	1.50381	48.58
93.1	1.50187	1.50453	48.62
93.2	1.50258	1.50524	48.67
93.3	1.50329	1.50595	48.72
93.4	1.50401	1.50667	48.76
93.5	1.50472	1.50738	48.81
93.6	1.50543	1.50810	48.85
93.7	1.50615	1.50881	48.90
93.8	1.50686	1.50952	48.94
93.9	1.50757	1.51024	48.99
94.0	1.50829	1.51096	49.03
94.1	1.50900	1.51167	49.08
94.2	1.50972	1.51239	49.12
94.3	1.51044	1.51311	49.17
94.4	1.51115	1.51382	49.22
94.5	1.51187	1.51454	49.26
94.6	1.51258	1.51526	49.31
94.7	1.51330	1.51598	49.35
94.8	1.51402	1.51670	49.40
94.9	1.51474	1.51742	49.44
95.0	1.51546	1.51814	49.49
95.1	1.51617	1.51886	49.53
95.2	1.51689	1.51958	49.58
95.3	1.51761	1.52030	49.62
95.4	1.51833	1.52102	49.67
95.5	1.51905	1.52174	49.71
95.6	1.51977	1.52246	49.76
95.7	1.52049	1.52318	49.80
95.8	1.52121	1.52390	49.85
95.9	1.52193	1.52463	49.90
96.0	1.52266	1.52535	49.94
96.1	1.52338	1.52607	49.98
96.2	1.52410	1.52680	50.03
96.3	1.52482	1.52752	50.08
96.4	1.52555	1.52824	50.12
96.5	1.52627	1.52897	50.16
96.6	1.52699	1.52969	50.21
96.7	1.52772	1.53042	50.25
96.8	1.52844	1.53114	50.30
96.9	1.52917	1.53187	50.34
97.0	1.52989	1.53260	50.39
97.1	1.53062	1.53332	50.43
97.2	1.53134	1.53405	50.48
97.3	1.53207	1.53478	50.52
97.4	1.53279	1.53551	50.57
97.5	1.53352	1.53623	50.61
97.6	1.53425	1.53696	50.66
97.7	1.53498	1.53769	50.70
97.8	1.53570	1.53842	50.75
97.9	1.53643	1.53915	50.79
98.0	1.53716	1.53988	50.84
98.1	1.53789	1.54061	50.88
98.2	1.53862	1.54134	50.93
98.3	1.53935	1.54207	50.97
98.4	1.54008	1.54280	51.02
98.5	1.54081	1.54353	51.06
98.6	1.54154	1.54426	51.10
98.7	1.54227	1.54499	51.15
98.8	1.54300	1.54573	51.19
98.9	1.54373	1.54646	51.24
99.0	1.54446	1.54719	51.28
99.1	1.54519	1.54793	51.33
99.2	1.54593	1.54866	51.37
99.3	1.54666	1.54939	51.42
99.4	1.54739	1.55013	51.46
99.5	1.54813	1.55087	51.50
99.6	1.54886	1.55160	51.55
99.7	1.54960	1.55234	51.59
99.8	1.55033	1.55307	51.64
99.9	1.55106	1.55381	51.68
100.0	1.55180	1.55454	51.73

INDEX

	CHAPTER AND SECTION.	PAGE.
Abrastol	IX, 35, 36	128
Acetanilid	XXVIII, 4	292
and acetphenetidin, in mixtures	XXVIII, 11–13	294
caffein, in mixtures	XXVIII, 1, 2	291
sodium salicylate, in mixtures	XXVIII, 18, 19	297
caffein and quinin sulphate, in mixtures	XXVIII, 21	298
morphin sulphate, in mixtures	XXVIII, 27	299
Acetphenetidin (phenacetin)	XXVIII, 7	293
and acetanilid, in mixtures	XXVIII, 11–13	294–296
caffein, in mixtures	XXVIII, 5–7	293
salol, in mixtures	XXVIII, 14–17	296, 297
Acetyl value, of fats and oils	XXII, 31	250
Acidity, in cheese	XXI, 57	235
cream of tartar	XXVII, 11	281
distilled liquors	XVI, 7	181
prepared mustard	XXIII, 30	262
total, in coffees	XXV, 20	271
fruits and fruit products	XII, 9	154
tanning liquors	IV, 15, 16	45
volatile, in coffees	XXV, 21	271
water-soluble, of feeds	VII, 67	98
Acids, fixed, in tomato products	XIII, 26	164
vinegars	XVIII, 18	194
wines	XV, 26	178
insoluble, in fats and oils	XXII, 22	247
mineral, free, in fruits and fruit products	XII, 11	155
vinegars	XVIII, 26–28	196
soluble, in fats and oils	XXII, 21	249
total, in beers	XVII, 10	188
canned vegetables	XIII, 7	162
tomato products	XIII, 23	163
vinegars	XVIII, 17	194
wines	XV, 23	177
volatile, in beers	XVII, 11	188
canned vegetables	XIII, 8	162
fruits and fruit products	XII, 10	155
tomato products	XIII, 24	163
vinegars	XVIII, 19	194
wines	XV, 24, 25	177
Aeration method for ammonia in meat	XX, 7, 8	209, 210
Agar-agar	XII, 21, 22	156
Albumin, in milk	XXI, 6, 7	225, 226
wheat flour	XIV, 12	168

	CHAPTER AND SECTION.	PAGE.
Alcohol, in beers	XVII, 4	187
confectionery	VIII, 28	109
distilled liquors	XVI, 2–4	181
flavoring extracts	XIX, 2, 18, 41, 48, 50, 54	197, 199, 204–206
fruits and fruit products	XII, 2	153
vinegars	XVIII, 4	191
wines	XV, 4	173
extract, in spices	XXIII, 10	258
precipitate, in fruits and fruit products	XII, 18	156
vinegars	XVIII, 22	195
Aldehydes, in distilled liquors	XVI, 9, 10	182
total, in flavoring extracts	XIX, 22, 23, 38	200, 201, 203
Alkali, black, in irrigating water	III, 88, 89	40
salts, in soils	XXIX, 30	320
Alkalinity, in industrial water	III, 80–83	39
of ash, in cacao products	XXIV, 6, 7	265
coffees	XXV, 10	270
fruits and fruit products	XII, 7	154
saccharine products	VIII, 15, 16	105, 106
teas	XXVI, 8	273
tomato products	XIII, 18	163
vinegars	XVIII, 15	194
wines	XV, 18, 19	176
Aluminium, in waters	III, 44	29
oxid, in plants	II, 6	16
soils	XXIX, 15	315
Allihn's Method for dextrose	VII, 52, 53	90
Almond extract	XIX, 41–47	204, 205
Alum, in baking powder	XXVII, 20	283
cream of tartar	XXVII, 20	283
Amaranth	X, 5, 7, 15	133, 134, 138
Ammonia, in baking powders	XXVII, 27	284
meat and meat products	XX, 7, 8	209, 210
free and albuminoid, in waters	III, 10, 11	22
Anilin chlorid test for commercial invert sugar	VIII, 47, 48	112
Anise extract	XIX, 57	207
Annatto	X, 24	144
Antipyrin	XXVIII, 9	294
and caffein, in mixtures	XXVIII, 8, 9	293, 294
Arsenic, in baking powder	XXVII, 35	289
foods	XI, 1–4	147–149
mineral water	III, 64–67	37
oxid, total, in insecticides and fungicides	VI, 18, 19, 34, 35	56, 59, 60
water-soluble, in insecticides and fungicides	VI, 30, 31	59
total, in insecticides and fungicides	VI, 3–5, 27–29, 36, 37, 49, 57	53, 54, 58, 60, 62, 63

	CHAPTER AND SECTION.	PAGE.
Arsenious oxid, sodium acetate-soluble in insecticides and fungicides	VI, 9, 10	55
total, in insecticides and fungicides	VI, 6–8, 16, 17, 32, 33, 38, 50, 51	54–56, 59, 60, 63
water-soluble, in insecticides and fungicides	VI, 11, 12, 20–22, 52	55, 57, 63
Ash, in baking powders	XXVII, 21	283
beers	XVII, 16	188
cacao products	XXIV, 3	265
canned vegetables	XIII, 4	161
coffees	XXV, 7	269
dairy products	XXI, 2, 23, 34, 55	225, 230, 231, 235
distilled liquors	XVI, 6	181
flavoring extracts	XIX, 8, 27	198, 202
foods and feeding stuffs	VII, 4	71
fruits and fruit products	XII, 6	154
leathers	V, 3, 4	49
meat and meat products	XX, 3	209
plants	II, 2	15
prepared mustard	XXIII, 26	262
saccharine products	VIII, 10–12	105
spices and other condiments	XXIII, 3	257
teas	XXVI, 5	273
vinegars	XVIII, 13	194
wheat flour	XIV, 2	167
wines	XV, 16	176
Atropin, in tablets	XXVIII, 33	301
Babcock method for fat in dairy products	XXI, 13–15, 61	227, 228, 236
Bacteria, in tomato pulp, etc.	XIII, 30	165
Baking powders	XXVII	277
Baudouin test for sesame oil	XXII, 38	254
Beef fat, in lard	XXII, 40, 41	254, 255
Beers	XVII	187
Benzaldehyde, in almond extract	XIX, 42, 43	204
Benzoic acid	IX, 6–11	119–121
Beta-naphthol	IX, 34	127
Bordeaux mixture	VI, 40–44	60–62
with lead arsenate	VI, 53	63
Paris green	VI, 45–52	62, 63
Boric acid	IX, 15, 16	122, 123
in mineral water	III, 57	35
Bromin, in mineral water	III, 57	35
Brown phase reaction for chlorophyll	X, 23	144
Butter	XXI, 40–50	232–234
renovated	XXI, 51, 52	234
Butyric acid, in tomato products	XIII, 25	164

	CHAPTER AND SECTION.	PAGE.
Cacao products	XXIV	265
Caffein, in coffees	XXV, 14, 15	270
drugs	XXVIII, 1–3, 5, 6, 8, 10, 21–30	291–294, 299, 300
teas	XXVI, 14	274
and acetanilid, in mixtures	XXVIII, 1–3	291, 292
acetphenetidin, in mixtures	XXVIII, 5, 6	293
antipyrin, in mixtures	XXVIII, 8, 10	293
acetanilid and codein sulphate, in mixtures	XXVIII, 24–26	294
quinin sulphate, in mixtures	XXVIII, 21–23	298, 299
quinin sulphate and morphin sulphate, in mixtures	XXVIII, 27–30	229, 300
Calcium, in mineral water	III, 45	29
plants	II, 7, 8	17
soils	XXIX, 16	315
arsenate	VI, 36	60
oxid, in ash of spices	XXIII, 6	257
Canned vegetables	XIII	161
Capsicum, in ginger extract	XIX, 53	206
Caramel, in distilled liquors	XVI, 23	186
Carbon, carbonate, in soils	XXIX, 5, 6	310, 311
inorganic, in soils	XXIX, 5, 6	310–312
plants	II, 4	15
organic, in soils	XXIX, 7, 10	312–314
dioxid, in insecticides and fungicides	VI, 41, 42, 46, 54	61–63
mineral water	III, 38	28
plant ash	II, 3	15
available, in baking powders	XXVII, 10	281
residual, in baking powders	XXVII, 9	281
total, in baking powders	XXVII, 2–8	277–281
Carbonic and bicarbonic acids, in waters	III, 39	28
Casein, in dairy products	XXI, 4, 5, 44	225, 233
milk chocolate	XXIV, 16	267
Cassia extract	XIX, 48, 49	205
Cereal foods	XIV	167
Cheese	XXI, 53–61	234–236
Chicory, in coffees	XXV, 24	272
Chlorids, in meat extracts	XX, 42	221
wines	XV, 22	176
Chlorin, in butter	XXI, 44	233
cyanids	VI, 60	64
plants	II, 15–17, 20	19, 20
water	III, 18, 19, 36, 71	24, 27, 38
wheat flour	XIV, 17, 18	169
Chlorophyll	X, 23	144
Cholesterol and phytosterol, in mixtures of animal and vegetable fats	XXII, 32, 33	251, 252
Cinnamon extract	XIX, 48, 49	205
Citral, in flavoring extracts	XIX, 24, 25, 35–37	201, 203
Citric acid, in fruits and fruit products	XII, 30, 31	159
Clarifying reagents for use in optical methods	VII, 13	74

	CHAPTER AND SECTION.	PAGE.
Clove extract	**XIX, 48, 49**	**205**
Coal tar dyes	**X, 2**	**131**
Coating and glazing substances, in coffees	**XXV, 22**	**271**
Cochineal	**X, 26**	**144**
Codein sulphate	**XXVIII, 26**	**299**
Coffee, green	**XXV, 1, 2**	**269**
roasted	**XXV, 3, 4**	**269**
Coffees	**XXV**	**269**
Color, in beers	**XVII, 2**	**187**
potable water	**III, 3, 4**	**21**
vinegars	**XVIII, 20**	**195**
changes produced with acids and alkalies	**X, 13**	**136**
residual, in vanilla extract	**XIX, 14**	**199**
value, in vanilla extract	**XIX, 13**	**199**
wheat flour	**XIV, 21**	**171**
water-insoluble, in whiskies	**XVI, 21**	**186**
Coloring matter and tannin, in wines	**XV, 29, 30**	**179**
Coloring matters	**X**	**131**
in dairy products	**XXI, 21, 29, 50, 58**	**229, 231, 234, 235**
wines	**XV, 35**	**180**
natural	**X, 16–26**	**139–144**
separation of, by immiscible solvents	**X, 4, 5**	**133**
Colors, insoluble in amyl alcohol in distilled liquors	**XVI, 22**	**186**
vanilla extract	**XIX, 15**	**199**
Condensed milk, sweetened	**XXI, 32–39**	**231, 232**
unsweetened	**XXI, 31**	**231**
Condiments, spices and other	**XXIII**	**257**
Copper, in foods	**XI, 8**	**151**
insecticides and fungicides	**VI, 43, 44, 47, 48**	**61–63**
carbonate	**VI, 39**	**60**
oxid, total, in insecticides and fungicides	**VI, 13, 14, 39**	**55, 56, 60**
reducing substances in spices	**XXIII, 12**	**258**
Cottonseed oil	**XXII, 36**	**253**
Coumarin, in vanilla extract	**XIX, 4, 5**	**197**
***Cowles method* for malic acid value in maple products**	**VIII, 64**	**114**
Cream	**XXI, 22–30**	**230, 231**
of tartar, in wines	**XV, 28**	**178**
baking powders	**XXVII, 16**	**282**
Creatin, in meat and meat products	**XX, 29, 52**	**215, 223**
Creatinin, in meat extracts	**XX, 53**	**223**
Crude fiber, in cacao products	**XXIV, 9**	**265**
coffees	**XXV, 16**	**271**
foods and feeding stuffs	**VII, 65, 66**	**97, 98**
prepared mustard	**XXIII, 32**	**262**
spices and other condiments	**XXIII, 14**	**258**
teas	**XXVI, 12**	**274**
wheat flour	**XIV, 4**	**167**
Cyanids, sodium and potassium	**VI, 59, 60**	**63, 64**

	CHAPTER AND SECTION.	PAGE.
Hardness, permanent, in industrial water	III, 86	40
temporary, in industrial water	III, 79	39
total, in industrial water	III, 84, 85	39, 40
Hehner Method for formaldehyde	IX, 19	124
number of fats and oils	XXII, 22	247
Herzfeld Gravimetric Method for invert sugar	VII, 34, 35	81
Hide substance, in leathers	V, 14, 15	51
Hiltner Method for citral in lemon and orange oils	XIX, 37	203
Honey	VIII, 31–49	109–112
Hydrocyanic acid, in almond extract	XIX, 45, 46	205
formed by hydrolysis of glucosides in beans	VII, 68–70	98, 99
Hydrogen sulphid, in mineral water	III, 37	27
Immersion refractometer method for methyl alcohol, in distilled liquors	XVI, 18	184
Indigo Carmine	X, 5, 7, 15	113, 134, 139
Insecticides and fungicides	VI	53
Invert sugar, in foods and feeding stuffs	VII, 19–38	77–82
commercial, in honey	VIII, 45–48	112
Iodin, in mineral water	III, 57, 62, 63	35, 36
absorption number of fats and oils	XXII, 15–18	244–246
Iron, in mineral water	III, 42, 43	29
soils	XXIX, 15	315
and aluminium, in mineral water	III, 41–44	29
Jorissen Method for sucrol or dulcin	IX, 38	128
test for salicylic acid	IX, 3	118
Kissling Method for nicotin in tobacco and tobacco extract	VI, 64, 65	65
Kjeldahl Method for organic and ammoniacal nitrogen in fertilizers	I, 16–18	5, 6
Modified, for total nitrogen, in fertilizers	I, 24–26	7, 8
-Gunning-Arnold Method for organic and ammoniacal nitrogen, in fertilizers	I, 22, 23	7
Kleber Method for citral in lemon and orange oils	XIX, 35, 36	203
Koettstorfer number of fats and oils	XXII, 19, 20	246
Lactose, in cacao products	XXIV, 15	267
dairy products	XXI, 8–11, 36	226, 231
foods and feeding stuffs	VII, 45–47	
Leach Method for formaldehyde	IX, 20	124
Lead, in baking powders	XXVII, 28–34	285–288
arsenate	VI, 23–35	57–60
number, in maple products	VIII, 61–68	114
vanilla extract	XIX, 6	198
oxid, total, in insech	VI, 24–26	57, 58
precipitate, in vinegars	XVIII, 11	193
Leathers	V	49
Leffman and Beam Method for soluble volatile acids, in fats and oils	XXII, 26, 27	248

	CHAPTER AND SECTION.	PAGE.
Fertilizers	I	1
Fish oil, in the presence of vegetable oils	XXII, 42	256
Flavoring extracts	XIX	197
Flour, wheat	XIV	167
Fluoborates and fluosilicates	IX, 27–29	126
Fluorids	IX, 25, 26	125, 126
Food preservatives	IX	117
Foods and feeding stuffs	VII	71
Foreign fats containing tristearin, in lard	XXII, 40, 41	254, 255
Formaldehyde	IX, 17–24	123–125
solutions	VI, 68–71	66, 67
Formic acid	IX, 39–41	128, 129
in vinegars	XVIII, 21	195
Fruits and fruit products	XII	153
Fungicides	VI	53
Fufural, in distilled liquors	XVI, 11, 12	182, 183
Fusel oil, in distilled liquors	XVI, 13, 14	183
Galactan, in foods and feeding stuffs	VII, 64	97
Gasoline color value, in wheat flour	XIV, 21	171
Gelatin, as coating substance, in coffees	XXV, 23	272
in dairy products	XXI, 19	229
fruits and fruit products	XII, 20	156
meat and meat products	XX, 48, 49	222
Ginger, in ginger extract	XIX, 52	206
cold water extract	XXIII, 11	258
extract	XIX, 50–53	206
Glazing substances, in coffees	XXV, 22, 23	271, 272
Globulin, in wheat flour	XIV, 12	168
Glucose, in leathers	V, 8, 9	50, 51
commercial, in fruits and fruit products	XII, 16	155
saccharine products	VIII, 21, 22	107
wines	XV, 15	176
Gluten, in wheat flour	XIV, 15, 16	169
Glutenin, in wheat flour	XIV, 13	168
Glycerol, in beers	XVII, 15	188
flavoring extracts	XIX, 3, 19	197, 200
meat extracts	XX, 55	223
vinegars	XVIII, 5, 6	191, 192
wines	XV, 5–7	174
Glycogen, in meat	XX, 16–18	212, 213
Gum, in wines	XV, 33	179
Gunning Method for organic and ammoniacal nitrogen in fertilizers	I, 19–21	7
Modified, for total nitrogen in fertilizers	I, 27, 28	8
-Hibbard Method for total nitrogen in soils	XXIX, 11	314
Halphen test for cottonseed oil	XXII, 36	253
Hanus Method for iodin absorption number of fats and oils	XXII, 15, 16	244, 245
Harding-Parkin Method for fat in ice cream	XXI, 66	237

	CHAPTER AND SECTION.	PAGE.
Moisture, in Bordeaux mixture	VI, 40	60
Bordeaux mixture with lead arsenate	VI, 53	63
Paris green	VI, 45	62
cacao products	XIV, 2	265
canned vegetables	XIII, 3	161
coffees	XXV, 5	269
dairy products	XXI, 41, 54	232, 234
fertilizers	I, 3	1
foods and feeding stuffs	VII, 2, 3	71
insecticides and fungicides	VI, 2, 15, 23, 40, 45, 53, 61	53, 56, 57, 60, 62–64
lead arsenate	VI, 23	57
leathers	V, 2	49
London Purple	VI, 15	56
meat and meat products	XX, 2, 39	209, 221
Paris green	VI, 2	53
saccharine products	VIII, 2–4, 32, 51	101, 110, 113
soap	VI, 61	64
soils	XXIX, 3	309
spices	XXIII, 2	257
teas	XXVI, 3	273
wheat flour	XIV, 1	167
Molds, in tomato pulp, etc.	XIII, 28	164
Morphin sulphate	XXVIII, 30	300
Morpurgo Method for sucrol or dulcin	IX, 37	128
Munson and Walker Method for reducing sugars	VII, 24, 25	78
Mustard, prepared	XXIII, 24–34	261–263
Naphthol Yellow S	X, 5, 8, 15	133, 135, 139
Nitrate method for nitroglycerin in tablets	XXVIII, 37, 38	303
Nitrates, in meat and meat products	XX, 9–13	210, 211
wines	XV, 34	179
Nitrite method for nitroglycerin in tablets	XXVIII, 39, 40	303, 304
Nitrobenzol, in almond extract	XIX, 47	205
Nitrogen, in fertilizers	I, 16–39	5–11
leathers	V, 14	51
nitrate salts, in fertilizers	I, 35	10
non-volatile ether extract, in spices	XXIII, 8	257
saccharine products	VIII, 18	106
spices	XXIII, 7	257
albuminoid, in foods and feeding stuffs	VII, 6, 7	72
alcohol-soluble, in meat extracts	XX, 24, 25	214, 215
amido, in foods and feeding stuffs	VII, 8	72
amino, in meat	XX, 30–33	216–219
wheat flour	XIV, 12	168
ammoniacal, in fertilizers	I, 16–23	5–7
meat	XX, 7	209
coagulable, in meat	XX, 26	215
insoluble, in meat products	XX, 46	222
nitrate, in fertilizers, detection of	I, 15	5
waters	III, 14–17	23, 24

	CHAPTER AND SECTION.	PAGE.
Nitrogen, nitric and ammoniacal, in fertilizers	I, 33, 34	10
nitrite, in waters	III, 12, 13	23
wheat flour	XIV, 19, 20	170
organic and ammoniacal, in fertilizers	I, 16–23	5–7
proteose, peptone and gelatin, in meat	XX, 27	215
soluble and insoluble, in meat	XX, 24, 25	214, 215
total, in cacao products	XXIV, 8	265
water-insoluble, soluble in neutral permanganate	I, 36, 37	11
distilled from alkaline permanganate	I, 38, 39	11
total, in dairy products	XXI, 3, 56	225, 235
fertilizers	I, 24–31	7–9
meat and meat products	XX, 6, 44	209, 222
soils	XXIX, 11, 12	314
spices	XXIII, 7, 8	257
Nitroglycerin, in tablets	XXVIII, 36–40	302–304
Non-tannins, in tanning materials	IV, 6, 7	44
Nutmeg extract	XIX, 57	207
Odor, in potable water	III, 5	21
Oil, in anise and nutmeg extracts	XIX, 57	207
cassia, cinnamon and clove extracts	XIX, 49	205
peppermint, spearmint and wintergreen extracts	XIX, 55	206
-soluble dyes	X, 3	132
Oleomargarine	XXI, 51, 52	234
Olive oil, in paprika	XXIII, 18	259
Optical rotation, of lemon and orange oils	XIX, 34	202
Orange extract	XIX, 17–31	199–202
oil	XIX, 32–40	202, 203
in orange extract	XIX, 20, 21	200
Oxygen, dissolved, in potable water	III, 24–29	26, 27
required, in potable water	III, 20–23	25
Paraffin, in confectionery	VIII, 27	109
teas	XXVI, 18	275
Paris green	VI, 2–14	53–56
Peanut oil	XXII, 37	253
Pentosans, in foods and feeding stuffs	VII, 62, 63	96
vinegars	XVIII, 23	195
wines	XV, 32	179
Peppermint extract	XIX, 54, 55	206
Pepsin, in liquids	XXVIII, 41–43	304, 305
Petroleum ether extract, in coffees	XXV, 19	271
teas	XXVI, 10	274
Phloroglucol test for formaldehyde	IX, 24	125
Phosphoric acid, in baking powders	XXVII, 25	284
beers	XVII, 17	188
fertilizers	I, 4–14	1–5
mineral water	III, 55, 56	34
plants	II, 9–11	18

	CHAPTER AND SECTION.	PAGE.
Phosphoric acid, in *Thomas* or basic slag	I, 48–53	14
wines	XV, 20	176
soluble and insoluble, in vinegars	XVIII, 16	194
Phosphorus, in soils	XXIX, 21, 22	317, 318
soluble, in blood, brain and glandular organs	XX, 36, 37	219–221
total, soluble, in meat and meat products	XX, 34	219
total, in meat	XX, 5	209
Physical constants of the 10% distillate, in flavoring extracts	XIX, 39	203
examination, of canned vegetables	XIII, 1	161
coffees	XXV, 1, 3	269
vinegars	XVIII, 1	191
wines	XV, 1	173
Phytosterol, in mixtures of animal and vegetable fats	XXII, 32, 33	251, 252
Pigments	X, 1	131
used in teas for coloring or facing	XXVI, 19	275
Pinene, in lemon and orange oils	XIX, 40	203
Plant constituents	II	15
Polarization, in foods and feeding stuffs	VII, 14–17	74–76
fruits and fruit products	XII, 13	155
honey	VIII, 36, 37	110
maple products	VIII, 52, 54	113
saccharine products	VIII, 19, 20	106, 107
vinegars	XVIII, 12	193
direct, in beers	XVII, 14	188
method, for lemon and orange oils	XIX, 20	200
Polenske Method for insoluble volatile acids in fats and oils	XXII, 28	249
number of fats and oils	XXII, 28	249
Ponceau 3R	X, 5, 8, 15	133, 135, 138
Potash, in fertilizers	I, 40–45	12, 13
Potassium, in mineral water	III, 49–51	31–33
plants	II, 13, 14, 18	18, 19, 20
soils	XXIX, 23	318
bitartrate, in baking powders	XXVII, 16	282
Preparation of sample, in baking powders	XXVII, 1, 29	277, 285
cacao products	XXIV, 1	265
butter	XXI, 40, 46	232, 233
canned vegetables	XIII, 2	161
cheese	XXI, 53	234
coffees	XXV, 4	269
condensed milk	XXI, 32	231
drugs	XXVIII, 1, 5, 8, 18, 21, 27, 36	291, 293, 297–299, 302
fats and oils	XXII, 1	239
fertilizers	I, 2	1
foods and feeding stuffs	VII, 1	71
fruits and fruit products	XII, 1	153
honey	VIII, 31	109
ice cream	XXI, 64	236

	CHAPTER AND SECTION.	PAGE.
Preparation of sample, in insecticides and fungicides	VI, 1	53
leathers	V, 1	49
maple products	VIII, 50	113
meat and meat products	XX, 1, 38	209, 221
plants	II, 1	15
prepared mustard	XXIII, 24	261
preservatives	IX, 1, 6, 13, 17, 27	117, 119, 121, 123, 126
saccharine products	VIII, 1	101
soils	XXIX, 2	302
spices and other condiments	XXIII, 1	257
tanning materials	IV, 1, 11, 18	43, 45, 46
teas	XXVI, 2	273
tomato products	XIII, 12	162
vinegars	XVIII, 2	191
wines	XV, 2	173
Preservatives, in foods	IX	117
milk	XXI, 20	229
wines	XV, 36	180
Protein, in beers	XVII, 18	189
condensed milk	XXI, 35	231
fruits and fruit products	XII, 12	155
prepared mustard	XXIII, 29	262
teas	XXVI, 11	274
wheat flour	XIV, 7	167
crude, in foods and feeding stuffs	VII, 5	71
wines	XV, 31	179
Proteins, alcohol-soluble, in wheat flour	XIV, 8–10	167, 168
soluble in 5% potassium sulphate	XIV, 11	168
Proteoses, in meat and meat products	XX, 27, 48	215, 222
Quantitative method, for free mineral acids in vinegars	XVIII, 28	196
starch in meat	XX, 15	212
Quinin sulphate	XXVIII, 23	299
Raffinose, in beet products	VII, 17	76
Raw sugars, general directions for optical methods	VII, 12	73
Reduced iron method for nitric and ammoniacal nitrogen in fertilizers	I, 33	10
Reducing substances, total, before inversion, in beers	XVII, 12	188
vinegars	XVIII, 8	193
sugars, in beers	XVII, 12	188
foods and feeding stuffs	VII, 19–38	77–82
fruits and fruit products	XII, 15	155
saccharine products	VIII, 23, 38	107, 110

	CHAPTER AND SECTION.	PAGE.
Reducing sugars, in wines	XV, 12	175
after inversion, in tomato products	XIII, 21	163
vinegars	XVIII, 10	193
before inversion, in tomato products	XIII, 20	163
vinegars	XVIII, 9	193
other than dextrose, in foods and feeding stuffs	VII, 55	94
Refractive index, of fats and oils	XXII, 5–8	240, 241
lemon and orange oils	XIX, 33	202
Reichert-Meissl Method for soluble volatile acids in fats and oils	XXII, 23–25	247, 248
number, of fats and oils	XXII, 23–27	247, 248
Renovated butter	XXI, 51, 52	234
Residual color after precipitation with lead acetate, in flavoring extracts	XIX, 14	199
Resin oil	XXII, 35	253
Resorcin test for commercial invert sugar	VIII, 45, 46	112
Riche and Bardy Method for methyl alcohol in distilled liquors	XVI, 17	184
Roese-Gottlieb Method, for ether extract in confectionery	VIII, 26	108
fat in dairy products	XXI, 12	227

Saccharin	IX, 12–14	121, 122
Saccharine products	VIII	101
Salicylic acid	IX, 1–5	117–119
Salol, in drugs	XXVIII, 16, 17	297
and acetphenetidin in mixtures	XXVIII, 14, 15	296
Salt, in butter	XXI, 45	233
canned vegetables	XIII, 5	161
cheese	XXI, 55	235
prepared mustard	XXIII, 27	262
tomato products	XIII, 19	163
Sand, in plants	II, 4	15
tomato products	XIII, 16	163
Sanglé-Ferrière Method for abrastol	IX, 36	128
Santonin, in wormseed	XXVIII, 35	302
Saponification number of fats and oils	XXII, 19, 20	246
Schmidt-Bondzynski Method, Modified, for fat in cheese	XXI, 60	235
Serum, acetic, in milk	XXI, 16	228
copper, in milk	XXI, 18	229
sour, in milk	XXI, 17	229
Sesame oil	XXII, 38, 39	254
Silica, in mineral water	III, 40	28
plants	II, 4	15
Sinibaldi Method for abrastol	IX, 35	128
Slag, *Thomas* or basic	I, 46–53	13, 14
Soap	VI, 61, 62	64
Soda lye	VI, 63	64

	CHAPTER AND SECTION.	PAGE.
Sodium, in mineral waters	III, 49–51	31–33
plants	II, 13,14	18, 19
soils	XXIX, 23	318
chlorid, in tomato products	XIII, 19	163
salicylate	XXVIII, 18, 20	297, 298
Soils	XXIX	309
Solids, in dairy products	XXI, 1	225
flavoring extracts	XIX, 7, 26, 51	198, 202, 206
fruits and fruit products	XII, 3–5	153, 154
leathers	V, 10–12	51
prepared mustard	XXIII, 25	262
tanning materials	IV, 2–5, 12, 13	43–45
vinegars	XVIII, 7	193
waters	III, 6, 7, 31, 70	21, 27, 38
insoluble, in fruits and fruit products	XII, 4, 5	154
tomato products	XIII, 14	162
non-sugar, in wines	XV, 11	175
soluble, in coffees	XXV, 6	269
Sörenson Method for amino nitrogen, in meat	XX, 33	219
Soxhlet Method for dextrose	VII, 50	90
invert sugar	VII, 22, 23	78
-*Wein Method* for lactose	VII, 46, 47	88
Spearmint extract	XIX, 54, 55	206
Special tests for coal tar dyes permitted under the Federal Food and Drugs Act	X, 15	138
Specific gravity, of beers	XVII, 3	187
distilled liquors	XVI, 1	181
fats and oils	XXII, 2	239
flavoring extracts	XIX, 1, 17	197, 199
saccharine products	VII, 5, 7	102, 103
vinegars	XVIII, 3	191
wines	XV, 3	173
Spices	XXIII, 1–23	257–261
microscopic examination of	XXIII, 19–23	260, 261
and added pungent materials in vinegars	XVIII, 31	196
other condiments	XXIII	257
Spores, in tomato pulp, etc.	XIII, 29	165
Stahlschmidt Method, Modified, for caffein in coffees	XXV, 15	270
Starch, in baking powders	XXVII, 17–19	282, 283
cacao products	XXIV, 10, 11	266
coffees	XXV, 17	271
foods and feeding stuffs	VII, 59–61	95, 96
fruits and fruit products	XII, 19	156
meat	XX, 14, 15	212
saccharine products	VIII, 24	107
spices	XXIII, 13	258
Strontium, in mineral water	III, 46	30
Strychnin, in liquids	XXVIII, 31	300
tablets	XXVIII, 32	301
Sucrol	IX, 37, 38	128

	CHAPTER AND SECTION.	PAGE.
Sucrose, in beets	VIII, 66–68	115, 116
cacao products	XXIV, 15	267
condensed milk	XXI, 38, 39	231, 232
flavoring extracts	XIX, 10, 28	198, 202
foods and feeding stuffs	VII, 14–39, 58	74–83, 95
fruits and fruit products	XII, 13, 14	155
saccharine products	VIII, 19, 20, 39, 54, 55, 66–68	106, 107, 110, 113, 115, 116
tomato products	XIII, 22	163
wines	XV, 13, 14	175, 176
Sugar, as a coating and glazing substance, in coffees	XXV, 22	271
house products	VIII, 66–68	115, 116
Sugars, in canned vegetables	XIII, 6	162
distilled liquors	XVI, 15	184
meat and meat products	XX, 19, 20, 56	213, 224
wheat flour	XIV, 6	167
total, in cattle foods	VII, 56–58	94, 95
Sulphate sulphur, in insecticides and fungicides	VI, 81	69
Sulphid sulphur, in insecticides and fungicides	VI, 78–80	68, 69
Sulphite-cellulose, in tanning materials	IV, 9, 10	44
Sulphur, in mustard	XXIII, 16	259
plants	II, 19	20
soils	XXIX, 20	317
Sulphuric acid, in baking powders	XXVII, 26	284
mineral water	III, 48	31
plants	II, 12	18
wines	XV, 21	176
Sulphurous acid	IX, 30–33	126, 127
Sweetening substitutes in fruits and fruit products	XII, 35	160
Tannin, in cloves and allspice	XXIII, 15	259
leathers	V, 13	51
tanning materials	IV, 8	44
teas	XXVI, 15, 16	274
wines	XV, 29, 30	179
Tanning extracts	IV, 1–10	43–45
liquors	IV, 11–16	45
materials	IV	43
raw and spent	IV, 17–21	46, 47
Tartaric acid, free, in baking powders	XXVII, 14, 15	282
wines	XV, 28	178
or combined, in baking powders	XXVII, 12	281
total, in baking powders	XXVII, 13	281
fruits and fruit products	XII, 23, 24	156, 157
vinegars	XVIII, 25	195
wines	XV, 27	178
and tartrates, in vinegars	XVIII, 24	195
Tartrazine	X, 5, 7, 15	133, 134, 138
Teas	XXVI	273
Thiosulphate sulphur, in insecticides and fungicides	VI, 75–77	68

	CHAPTER AND SECTION.	PAGE.
Thomas or basic slag	I, 46–53	13, 14
Tin, in foods	XI, 5–7	149, 150
Titanic oxid, in soils	XXIX, 15	315
Titer test for fats and oils	XXII, 12, 13	242, 243
Tobacco and tobacco extract	VI, 64–67	65
Tomato products	XIII, 12–30	162–165
bacteria in	XIII, 30	165
molds in	XIII, 29	165
yeasts and spores in	XIII, 28	164
Total aldehydes, in flavoring extracts	XIX, 22, 23, 38	200, 201, 203
Tragacanth	XXVIII, 34	301
Trillat Method for methyl alcohol in distilled liquors	XVI, 16	184
Turbidity, in potable water	III, 1, 2	21
Turmeric	X, 25	144
Turpentine	XXVIII, 44–49	306, 307
Unsaponifiable residue, in fats and oils	XXII, 34	252
Vanilla extract and its substitutes	XIX, 1–16	197–199
resins	XIX, 11	198
Vanillin, in vanilla extract	XIX, 4, 5	197
Van Slyke Method for amino nitrogen in meat	XX, 30–32, 50	215, 223
Villavecchia Test for sesame oil	XXII, 39	254
Vinegars	XVIII	191
Volatile oil, in teas	XXVI, 13	274
Volumetric method for chlorin in plant ash	II, 16, 17	19
thiosulphate method for reduced copper	VIII, 27, 28	79
Water, added, in milk	XXI, 16–18	228, 229
extract, acidity of, in wheat flour	XIV, 5	167
in teas	XXVI, 4	273
industrial	III, 70–86	38–40
irrigating	III, 87–89	40
mineral	III, 30–69	27–38
potable	III, 1–29	21–27
Water-soluble material, in leathers	V, 6, 7, 11, 13	49–51
Waters	III	21
Waxes and fats, in coffees	XXV, 25	272
Wein Method for maltose	VII, 42, 43	86
Wijs Method for iodin absorption number, in fats and oils	XXII, 17, 18	245, 246
Wiley Method for melting point of fats and fatty acids	XXII, 9, 10	241
Wines	XV	173
Wintergreen extract	XIX, 54–56	206, 207
Winton lead number, in maple products	VIII, 61–63	114
Yeasts and spores, in tomato pulp, etc.	XIII, 29	165
Zinc, in foods	XI, 9	151
arsenite	VI, 37, 38	60

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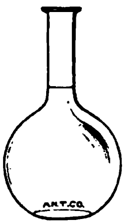

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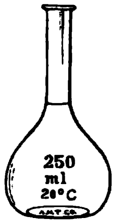

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